



UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of: **Stamm et al**

Application No. **09/899,026**

Group Art Unit: **1615**

Filed: **July 6, 2001**

Examiner: **H. Sheikh**

For: **Fenofibrate Pharmaceutical Composition Having High  
Bioavailability and Method for Preparing It**

Docket No: **107664.115US3**

Commissioner for Patents  
PO Box 1450  
Alexandria, VA 22313-1450

**Declaration under 37 CFR § 1.132 by Pascale Blouquin**

I, Pascale Blouquin, declare:

1. I am Head of Formulation Development at Laboratoires Fournier, SA, the assignee of US Application No. 09/899,026 (hereafter "the above-identified application").

2. To the best of my knowledge, understanding and belief, Laboratoires Fournier, SA, is the assignee of the above-identified application and of co-pending US Application Nos. 10/290,333, 10/665,520, 10/665,516, 10/665,519, 10/665,518, 10/665,517 and 10/665,522 (hereafter "the co-pending applications").

3. To the best of my knowledge, understanding and belief, the specification of the above-identified application is the same as the specification of the co-pending applications.

4. To the best of my knowledge, understanding and belief, Laboratoires Fournier's Lab Notebook No. 1 (attached as Exhibit 1) and Laboratoires Fournier's Lab Notebook No. 2 (attached as Exhibit 2) were transmitted around June 2004 to the counsel for Laboratoires Fournier at its request during document production for pending litigations related to Laboratoires Fournier's issued fenofibrate patents. This was the first time I had been asked to transmit to the counsel of Laboratoires Fournier these Lab Notebooks.

5. A certified English language translation of Lab Notebook No. 1 is attached as Exhibit 3; and a certified English language translation of Lab Notebook No. 2 is attached as Exhibit 4.

7. I have read and understand the contents of Lab Notebook Nos. 1 and 2.
8. Lab Notebook No. 1 covers the time period of 18 February 1997 to 15 May 1997.
9. Lab Notebook No. 2 covers the time period of 16 May 1997 to 30 July 1997.
10. To the best of my knowledge, understanding and belief, Lab Notebook Nos. 1 and 2 contain dissolution data of experiments conducted for the scale-up and development of the fenofibrate compositions of the invention described in the above-identified application and the co-pending applications.
11. To the best of my knowledge, understanding and belief, Lab Notebook Nos. 1 and 2 also contain dissolution data of capsules comprising 200 mg fenofibrate in various dissolution media.
12. The Examiner's attention is specifically directed to the dissolution data of Commercial Lot No. 2177 as follows:
  - (a) Example 2 and Figures 1 and 2 in the above-identified application and the co-pending applications provide the dissolution profile for a capsule comprising 200 mg fenofibrate from Commercial Lot No. 2177 in a dissolution medium constituted by water with 2% by weight Polysorbate 80 as measured using the rotating paddle method at 75 rpm, using the European Pharmacopoeia paddle apparatus.
  - (b) Lab Notebook No. 1 at Fournier Bates No. 1001569 provides the dissolution data for a capsule comprising 200 mg fenofibrate from Commercial Lot No. 2177 in a dissolution medium constituted by water with 0.025 M sodium lauryl sulfate as measured using the rotating paddle method at 75 rpm, using the European Pharmacopoeia paddle apparatus.



Declaration under 37 CFR § 1.132 of Pascale Blouquin

US Application No. 09/899,026

Page 3 of 5

These results of Commercial Lot No. 2177 are shown in Table 1 below.

**Table 1**

	5 minutes	10 minutes	20 minutes	30 minutes	60 minutes	120 minutes
Lot No. 2177 in Above- Identified Application and the Co-Pending Applications in Example 2 in dissolution medium constituted by water with 2% by weight Polysorbate 80	0%	3.7%	16.5%	54.9%	not measured	not measured
Lot No. 2177 in Lab Notebook 1 at Fournier Bates No. 1001569 in a dissolution medium constituted by water with 0.025 M sodium lauryl sulfate	1.6%	19.7%	55.5%	67.7%	78.0%	85.5%

13. A summary of the dissolution data of the capsules comprising 200 mg fenofibrate from Lab Notebook Nos. 1 and 2 is shown in Table 2 below. The dissolution medium used are shown in Table 3.

**Table 2**

Fournier Bates No.	Dissolution Medium	5 min.	10 min.	20 min.	30 min	60 min	120 min
1001532	A	no data	26.8	46.4	56.5	no data	no data
1001532	B	6.5	24.7	43.4	53.3	64.4	75.7
1001532	C	7.1	30.2	52.2	62.9	75.9	83.5
1001560	C	3.4	19.1	52.0	65.7	78.5	86.8
1001562	C	0.8	9.8	46.8	63.0	79.7	88.4
1001566	C	1.8	13.5	50.1	66.0	82.3	88.3
1001569	C	1.6	19.7	55.5	67.7	78.0	85.5
1001653	C	4.8	23.1	55.4	66.8	no data	88.0
1001662	C	2.9	20.2	54.3	66.7	79.4	87.2
1001672	C	3.7	21.7	54.3	66.3	80.1	88.1
1001679	C	5.1	28.5	57.0	68.2	80.8	88.2
1001689	C	5.3	29.8	55.4	65.6	75.7	84.5

Fournier Bates No.	Dissolution Medium	5 min.	10 min.	20 min.	30 min	60 min	120 min
1001692	C	4.9	28.2	57.1	67.0	79.0	83.8
1001703	C	1.3	11.4	50.6	63.4	76.2	84.9
1001707	C	4.4	21.7	53.5	66.0	79.4	87.9
1001711	C	4.6	22.5	55.3	66.8	80.3	87.9
1001715	C	4.6	24.3	54.9	66.2	78.4	87.8
1001849	D	no data	26.3	76.1	88.0	98.4	102.6

Table 3

Dissolution Medium	Characteristic of Dissolution Medium
A	dissolution medium constituted by water with 2% by weight Polysorbate 80 using the rotating paddle method at 75 rpm according to the European Pharmacopoeia
B	dissolution medium constituted by water with 0.02 M sodium lauryl sulfate using the rotating paddle method at 75 rpm according to the European Pharmacopoeia
C	dissolution medium constituted by water with 0.025 M sodium lauryl sulfate using the rotating paddle method at 75 rpm according to the European Pharmacopoeia
D	dissolution medium constituted by water with 0.1 M sodium lauryl sulfate using the rotating paddle method at 90 rpm according to the European Pharmacopoeia

14. In my opinion, the claimed invention in the above-identified application and the copending applications has superior properties when compared to the dissolution data in the Laboratory Notebooks submitted herewith. A comparison of the pending claims, the Inventive Example 2 in the present application, the comparative Example 2 in the present application that is Lipanthyl® 200M from Lot No. 2177, and the dissolution data in the attached Laboratory Notebook No. 1 at Bates Number Fournier 1001569, which is also representative of Lipanthyl® 200M from Lot No. 2177, is set forth in the table below.

Time	% Dissolution Recited in Pending Claims	% Dissolution by Inventive Example 2 in the Application	% Dissolution of Lipanthyl® 200M from Lot No. 2177 in Lab Notebook No. 1 at Fournier No. 1001569	% Dissolution of Lipanthyl® 200M from Lot No. 2177 in Example 2 in the Application
30 minutes	at least 75	95.9	67.7	54.9
60 minutes	--	--	78.0	--

15. The claimed invention requires at least 75% dissolution in 30 minutes. The data in the attached Laboratory Notebook No. 1 shows that it takes almost 60 minutes for the Lipanthyl® 200M, to achieve a dissolution of 78%. In other words, it takes almost twice as long for Lipanthyl® 200M to achieve a dissolution that the claimed invention requires in 30 minutes. In view of these results, it is my opinion that the dissolution profile of the inventive example 2 in the Application is clearly faster than the dissolution profile of Lipanthyl® 200M.

16. I was also asked to review Quality Assurance documents (attached as Exhibit <sup>5</sup> / b) that are analysis certificates of different batches of product. In the analysis certificates, dissolution results are mentioned. The dissolution was done in 0.1 M SLS. The dissolution of fenofibrate in 0.1M SLS is much more faster than the dissolution in 0.025 M SLS, as the 0.1M SLS medium contains 4 times more SLS than a 0.025 SLS medium, and furthermore was done at higher speed.

17. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements so made are punishable by fine or imprisonment or both under § 1001 of Title 18 of the United States Code and that such willful statements may jeopardize the validity or enforceability of the present application or any patent issued thereon.

  
\_\_\_\_\_  
Pascale Blouquin

Feb, 17<sup>th</sup> 2005  
\_\_\_\_\_  
Date

exh. 1

**CAHIER LF 178TER  
DISSOLUTION N°1**

FOURNIER 1001439

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BEST AVAILABLE COPY

Service Galénique

- Cahier Dissolution N° 1 -

- L F 178 TER -

Cahier approuvé le 26/04/00 par stb  
stb 26/04/00.

Cahier commencé le 18.02.97 -  
terminé le 15 Nov 97

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18.02.97

Essais Dissolution  
Tween 80 à 2%

I Speches - Gamme étalon

A. Préparation solution Tween 80 à 2%

Mettre 40 ml pipette graduée de Tween 80 ProLabo code 28 830 291 lot 045 FC dans une fiole jaugée de 2000 ml, faire le qs avec de l'eau purifiée du jour.

B. Préparation de la gamme étalon

Avec du comiconisat Fenofibrate/lauryl sulfate de sodium - ABB 1709.

Peser le comiconisat dans fiole jaugée de 200 ml faire le qs avec la solution de Tween 80 à 2%. Réaliser la gamme en double avec concentration en fenofibrate de 200 - 150 - 100 - 50 mg/l.

Pesée Balance GAL 205 - AG 204 -

Ultra-sons GAL 212 -

Agitation magnétique GAL 170.

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Après la mise en solution - 15 min ultra sons -  
mettre sous agitation magnétique une nuit -

Concentration en mg/l: 200 mg/l 150 mg/l - 100 mg/l - 50 mg/l  
en fenofibrate

Pesée Comiconisat 207 mg 155,25 mg 103,5 mg - 51,75 mg/l  
6/3/97

# C. Balayage Spectral

Uvikon 930 - GAL 108 Procédure: GAL051 GAL002 UFR03

Sauvegarde ligne de base = disquette Produit lip200cp N°2.

eau / eau : LBEAU

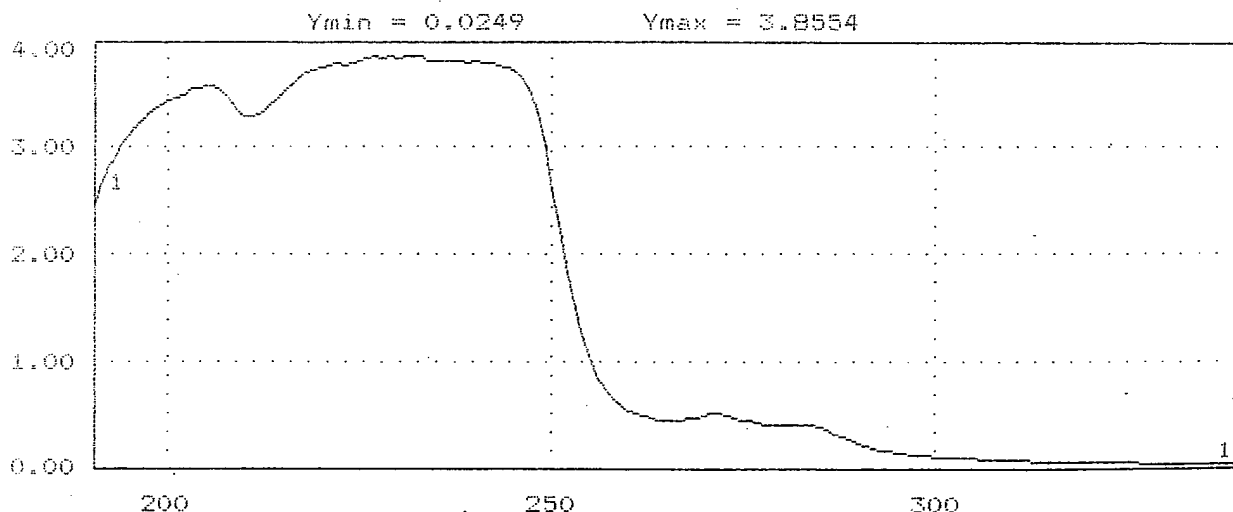
Tween 80 2% / Tween 80 2% : LBTWEEN.

Trace spectre ref: eau

ech: Tween 80 2%

BALAYAGE SPECTRAL EAU/TWEEN 80

18-02-1996 16:13



CONTRON INSTRUMENTS

UVIKON 930

AL

Données  
correspondantes:

$\lambda$ nm	do
300.0	41 0.1037_1
299.0	42 0.1093_1
298.0	43 0.1152_1
297.0	44 0.1232_1
296.0	45 0.1321_1
295.0	46 0.1442_1
294.0	47 0.1577_1
293.0	48 0.1739_1
292.0	49 0.1932_1
291.0	50 0.2151_1
290.0	51 0.2393_1
289.0	52 0.2666_1
288.0	53 0.2959_1
287.0	54 0.3264_1
286.0	55 0.3559_1
285.0	56 0.3812_1
284.0	57 0.3986_1
283.0	58 0.4094_1
282.0	59 0.4122_1
281.0	60 0.4106_1
280.0	61 0.4097_1

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RL

19.01.97.

Les solutions préparées le 18.02.97 pour la gamme étalon, sont inutilisables pour celle-ci, en effet les quantités pesées sont pour 1000 ml et non 200 ml, erreur d'un facteur 5.

Par contre ces solutions peuvent être utilisées pour déterminer la concentration à saturation du fenofibrate dans la solution de Tween 80 à 20%.

18.02.97 14:07:21

Code AM1709-1-200

Solution A

0.0000 g

85.5570 gT

0.0000 g

0.2070 gN

0.0000 g

85.7650 gB

Code AM 1709-2-200

Solution B.

0.0000 g

85.7492 gT

0.0000 g

0.2073 gN

0.0000 g

85.9570 gB

HC

Concentration solution A en fenofibrate

$$\frac{207 \times 200 \times 1000}{207 \times 200} = 1000 \text{ mg/l}$$

Concentration solution B en fenofibrate

$$\frac{207,3 \times 200 \times 1000}{207 \times 200} = 1001,4 \text{ mg/l}$$

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Réalisation de spectres des 2 solutions filtrées contre l'eau et contre le Tween 80 2%.

Filtres utilisés: filtre Millex HA référence SL HA 025 NB

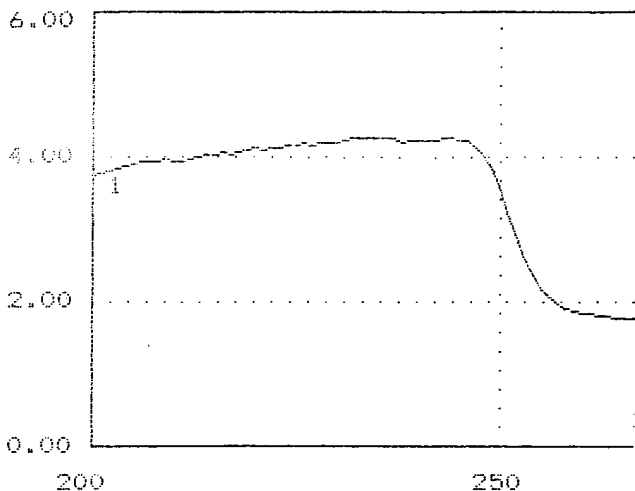
Les CS seront calculées après obtention de la gamme étalon. 8/3/97



# SOLUTION A CONTRE EAU

19-02-1996 14:26

Ymin = 0.0743 Ymax = 4.2587



300.0	41	1.2984_1
299.0	42	1.3643_1
298.0	43	1.4256_1
297.0	44	1.4829_1
296.0	45	1.5364_1
295.0	46	1.5806_1
294.0	47	1.6231_1
293.0	48	1.6611_1
292.0	49	1.6968_1
291.0	50	1.7329_1
290.0	51	1.7692_1
289.0	52	1.8041_1
288.0	53	1.8373_1
287.0	54	1.8638_1
286.0	55	1.8850_1
285.0	56	1.8964_1
284.0	57	1.8997_1
283.0	58	1.8968_1
282.0	59	1.8866_1
281.0	60	1.8742_1
280.0	61	1.8671_1

A/EAU

CONTRON INSTRUMENTS

FOURNIER 1001444

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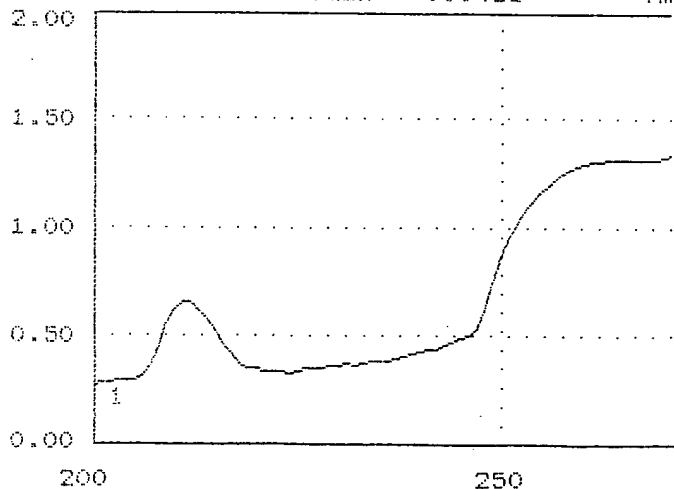
300.0	41	1.1947_1
299.0	42	1.2553_1
298.0	43	1.3103_1
297.0	44	1.3601_1
296.0	45	1.4036_1
295.0	46	1.4383_1
294.0	47	1.4664_1
293.0	48	1.4891_1
292.0	49	1.5061_1
291.0	50	1.5208_1
290.0	51	1.5303_1
289.0	52	1.5385_1
288.0	53	1.5439_1
287.0	54	1.5409_1
286.0	55	1.5341_1
285.0	56	1.5206_1
284.0	57	1.5067_1
283.0	58	1.4907_1
282.0	59	1.4793_1
281.0	60	1.4670_1
280.0	61	1.4630_1

A/Tween 80

:38

## SOLUTION A CONTRE TWEEN 80

Ymin = 0.0486 Ym



300.0	41	1.1947_1
299.0	42	1.2553_1
298.0	43	1.3103_1
297.0	44	1.3601_1
296.0	45	1.4036_1
295.0	46	1.4383_1
294.0	47	1.4664_1
293.0	48	1.4891_1
292.0	49	1.5061_1
291.0	50	1.5208_1
290.0	51	1.5303_1
289.0	52	1.5385_1
288.0	53	1.5439_1
287.0	54	1.5409_1
286.0	55	1.5341_1
285.0	56	1.5206_1
284.0	57	1.5067_1
283.0	58	1.4907_1
282.0	59	1.4793_1
281.0	60	1.4670_1
280.0	61	1.4630_1

CONTRON INSTRUMENTS

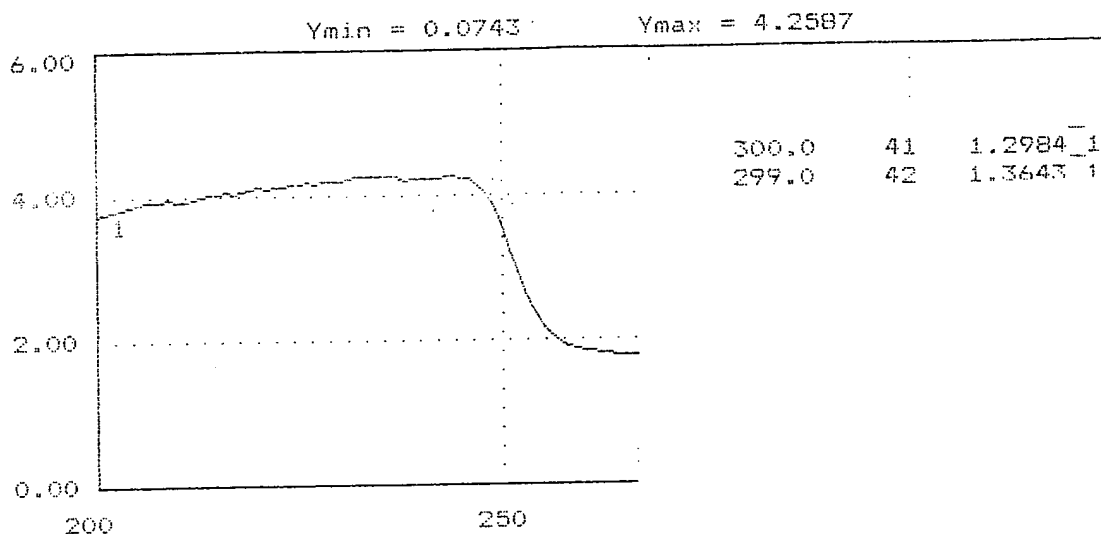
6/3/97

UVIKON 930

607

SOLUTION A CONTRE EAU

19-02-1996 14:26

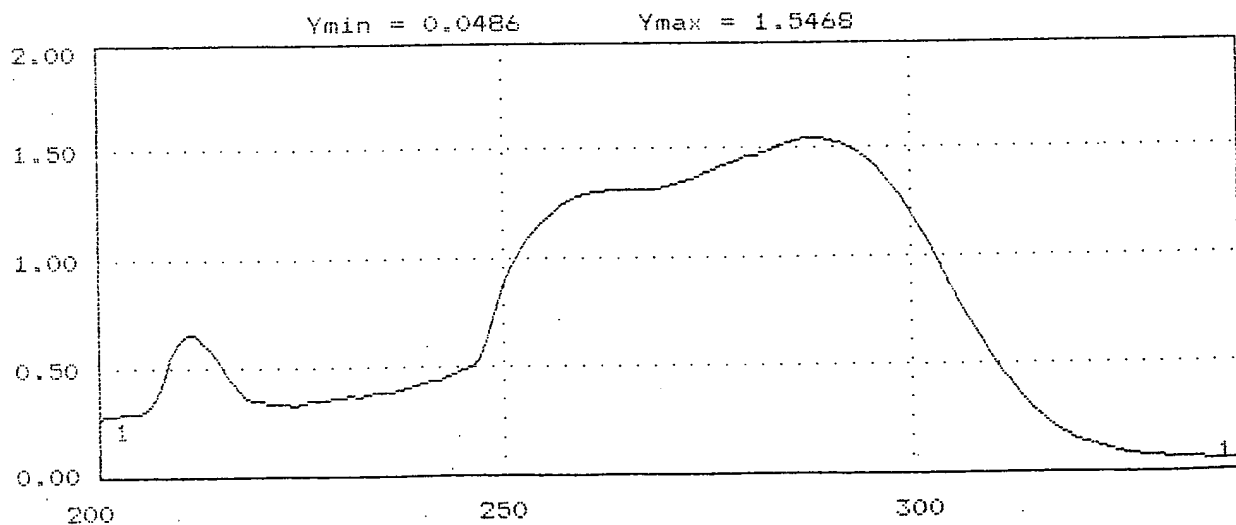


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SOLUTION A CONTRE TWEEN 80

19-02-1996 14:38



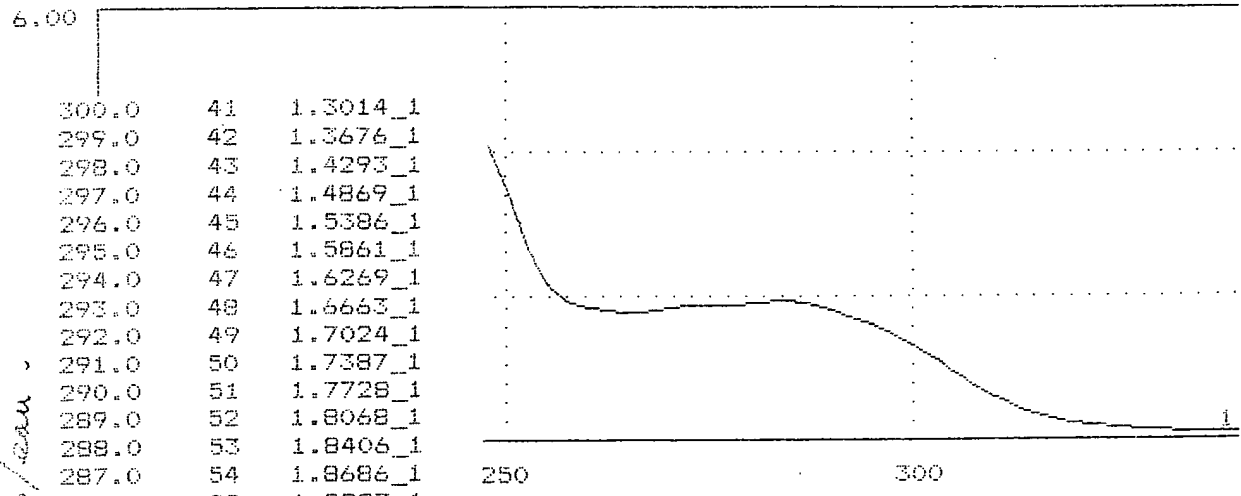
CONTRON INSTRUMENTS

UVIKON 930

SOLUTION B CONTRE EAU

19-02-1996 14:54

Ymin = 0.0739 Ymax = 4.3376



UVIKON 930

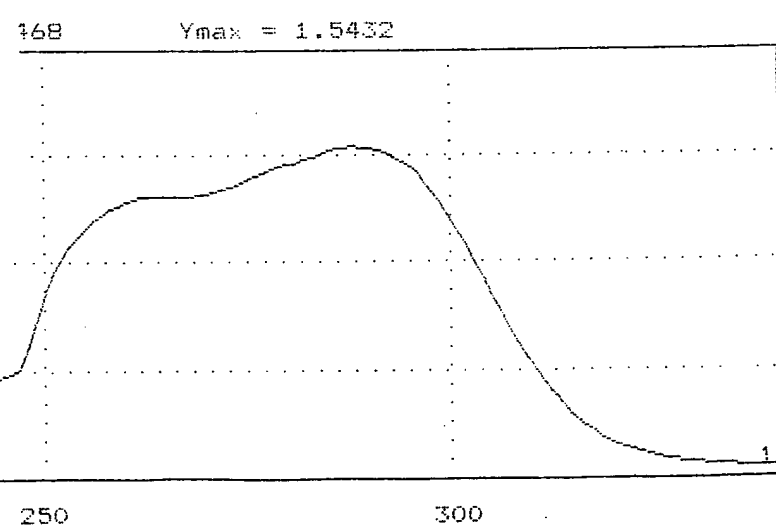
FOURNIER 1001446

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C/eau 80

299.0	42	1.2600_1
298.0	43	1.3143_1
297.0	44	1.3631_1
296.0	45	1.4075_1
295.0	46	1.4417_1
294.0	47	1.4679_1
293.0	48	1.4898_1
292.0	49	1.5067_1
291.0	50	1.5213_1
290.0	51	1.5303_1
289.0	52	1.5369_1
288.0	53	1.5403_1
287.0	54	1.5379_1
286.0	55	1.5298_1
285.0	56	1.5180_1
284.0	57	1.5021_1
283.0	58	1.4868_1
282.0	59	1.4726_1
281.0	60	1.4610_1
280.0	61	1.4561_1

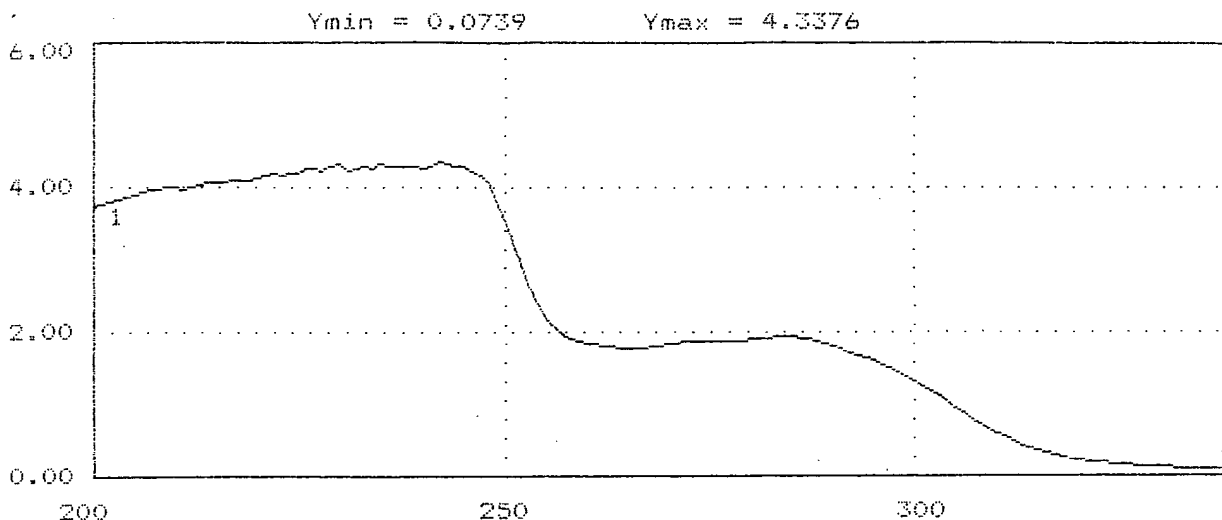
19-02-1996 14:47



007

SOLUTION B CONTRE EAU

19-02-1996 14:5



CONTRON INSTRUMENTS

UVIKON 93

FOURNIER 1001447

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19-02-1996 14:4

168 Ymax = 1.5432



CONTRON INSTRUMENTS

UVIKON 93

12 5/2/93

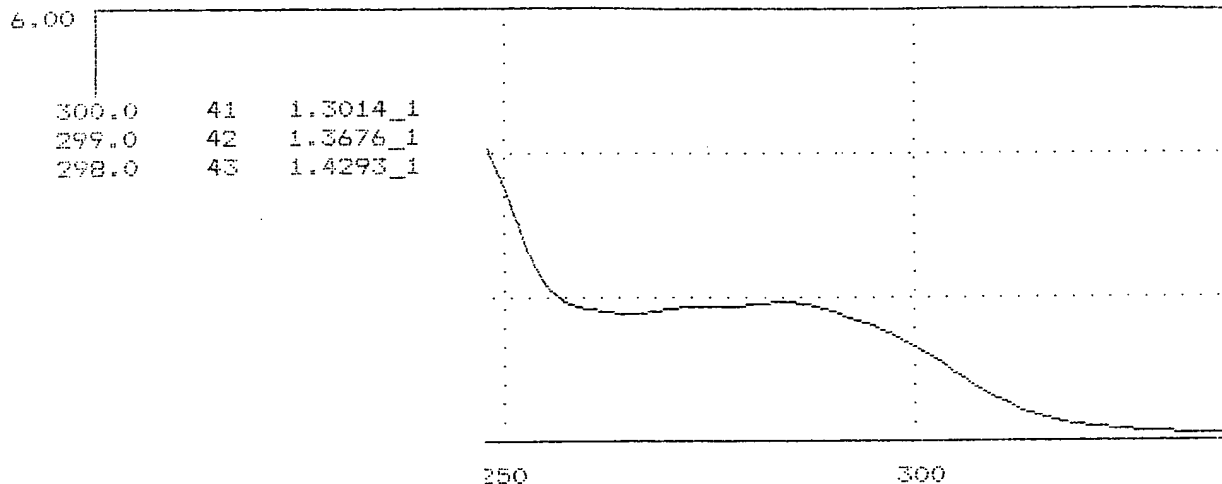
007

## SOLUTION B CONTRE EAU

19-02-1996 14:5

Ymin = 0.0739

Ymax = 4.3376



UVIKON 93

FOURNIER 1001448

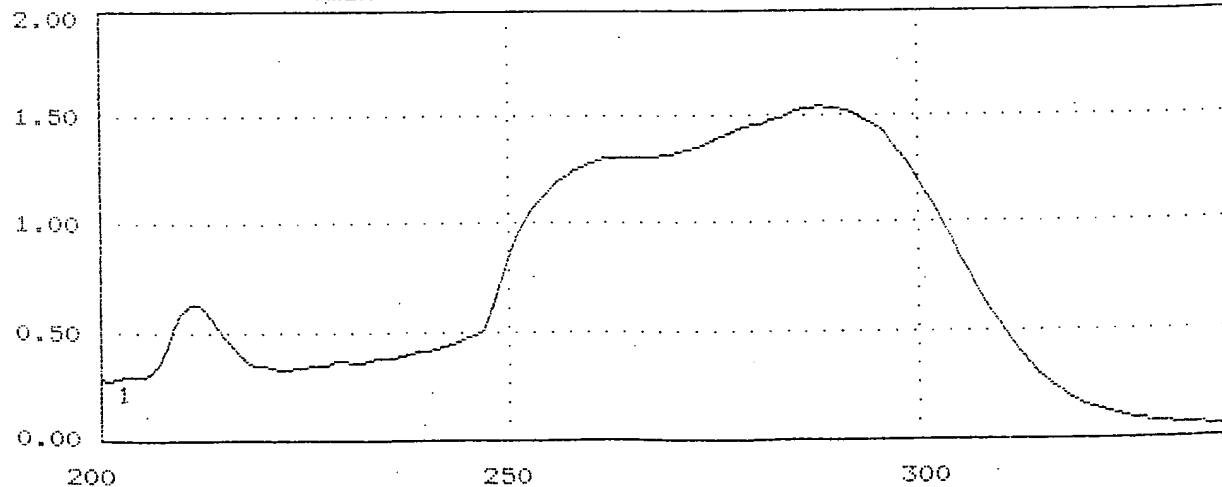
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## SOLUTION B CONTRE TWEEN 80

19-02-1996 14:4

Ymin = 0.0468

Ymax = 1.5432



CONTRON INSTRUMENTS

UVIKON 93

U.C.B.

19.02.97

## 2. Préparations de nouvelles solutions pour la gamme étalon.

matière première :

- comiconisat Féno/Lauryl Sulfate de Na : ARR 1709,

milieu utilisé :

- tween 80 à 2% Prolabo code 28830291 lot 045 FC

les pesées de comiconisat sont réalisées directement dans des fioles de 200 ml. le gsp 200 ml est effectué avec le tween 80 2%.

la gamme est préparée en double pour des concentrations de féno : 50 mg/l - 100 mg/l - 150 mg/l - 200 mg/l -

Utilisation de la balance AG204 GAL 205.

Concentration en féno fibre	Concentration en comiconisat	Pesée à réaliser en comiconisat pour un volume de 200 ml.
50 mg/l	51,75 mg/l	10,35 mg
100 mg/l	103,5 mg/l	20,7 mg
150 mg/l	155,25 mg/l	31,05 mg
200 mg/l	207 mg/l	41,4 mg

Utilisation du tween 80 à 2% préparé le 18/02/97 et préparation d'un nouveau litre de solution.

Introduction à la pipette graduée de 20 ml de tween 80 Prolabo code 28830291 dans une fiole jaugée de 1000 ml.

Qs à l'eau purifiée du jour. Agitation pendant 1 heure -

Les solutions sont laissées 1 nuit sous agitation -

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19.02.97 15:28:11  
Code 1709-41.4-1 sol A

0.0000 g  
79.9337 g T  
0.0000 g  
0.0417 g N  
0.0000 g  
-79.9755 g B

Code 1709-41.4-2 sol B

0.0000 g  
76.2448 g T  
0.0000 g  
0.0414 g N  
0.0000 g  
-76.2861 g B

Code 1709.3105-1 sol A

0.0000 g  
73.4429 g T  
0.0000 g  
0.0318 g N  
0.0000 g  
-73.4748 g B

Code 1709.3105-2 sol B

0.0000 g  
75.5857 g T  
0.0000 g  
0.0313 g N  
0.0000 g  
-75.5371 g B

ID 1709-20.7-1 sol A

0.0000 g  
73.5061 g T  
0.0000 g  
0.0218 g N  
0.0000 g  
-73.5271 g B

Code 1709-20.7-2 sol B

0.0000 g  
85.4478 g T  
0.0000 g  
0.0209 g N  
0.0000 g  
-85.4688 g B

Code 1709-1035-1 sol A

0.0000 g  
76.0366 g T  
0.0000 g  
0.0105 g N  
0.0000 g  
-76.0473 g B

Code 1709-1035-1 sol A

0.0000 g

Code 1709-1035-2 sol B

0.0000 g  
85.5346 g T  
0.0000 g  
0.0104 g N  
0.0000 g  
-85.5450 g B

Calcul des concentrations  
des solutions A et B.

### Solutions A

$$\begin{aligned} * 41,7 \times 5 \times 200 / 207 &= 201,45 \text{ mg/l} \\ * 31,8 \times 5 \times 150 / 155,25 &= 153,62 \text{ mg/l} \\ * 21,0 \times 5 \times 100 / 103,5 &= 101,45 \text{ mg/l} \\ * 10,5 \times 5 \times 50 / 51,75 &= 50,72 \text{ mg/l} \end{aligned}$$

### Solutions B

$$\begin{aligned} * 41,4 \times 5 \times 200 / 207 &= 200,00 \text{ mg/l} \\ * 31,3 \times 5 \times 150 / 155,25 &= 151,21 \text{ mg/l} \\ * 20,9 \times 5 \times 100 / 103,5 &= 100,97 \text{ mg/l} \\ * 19,4 \times 5 \times 50 / 51,75 &= 50,24 \text{ mg/l} \end{aligned}$$

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08-03/97.

20.02.97

[-] Réalisation de spectres de chaque solution contre l'eau et contre le tween 80.27.

Remarque: on utilisera les solutions à 50, 100 et 150 mg/l pour déterminer la gamme étalon; la solution à 200 mg/l servira à confirmer la concentration à saturation car elle n'est toujours pas limpide après 1 nuit d'agitation et 15 minutes d'ultrasons.

SOLUTION 50MG/L A CONTRE EAU

20-02-1996 09:58

Ymin = 0.0379

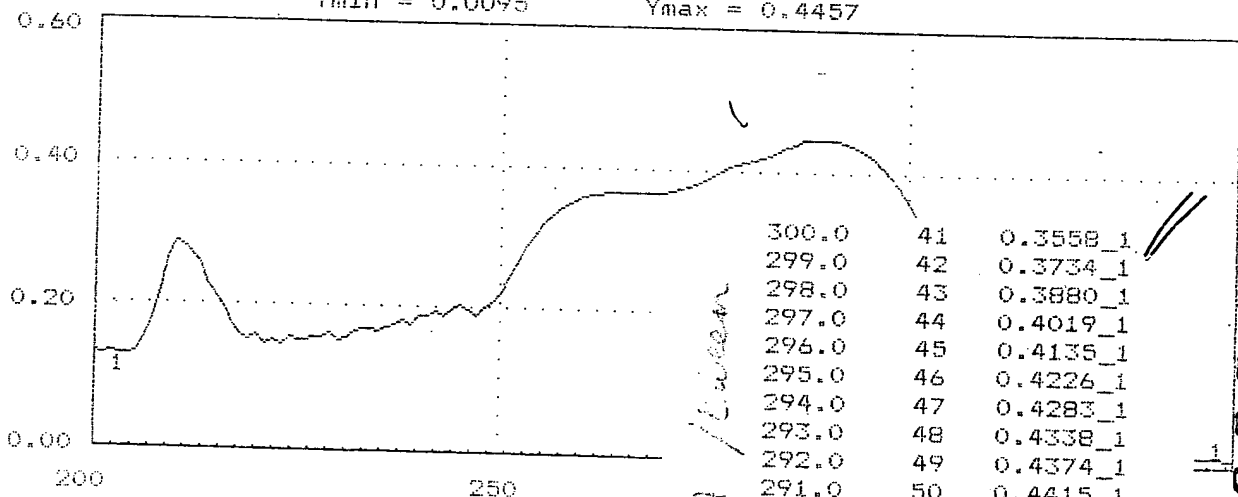
Ymax = 4.0540

SOLUTION 50MG/L A CONTRE TWEEN 80

20-02-1996 09:50

Ymin = 0.0095

Ymax = 0.4457



CONTRON INSTRUMENTS

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20 6/3/97



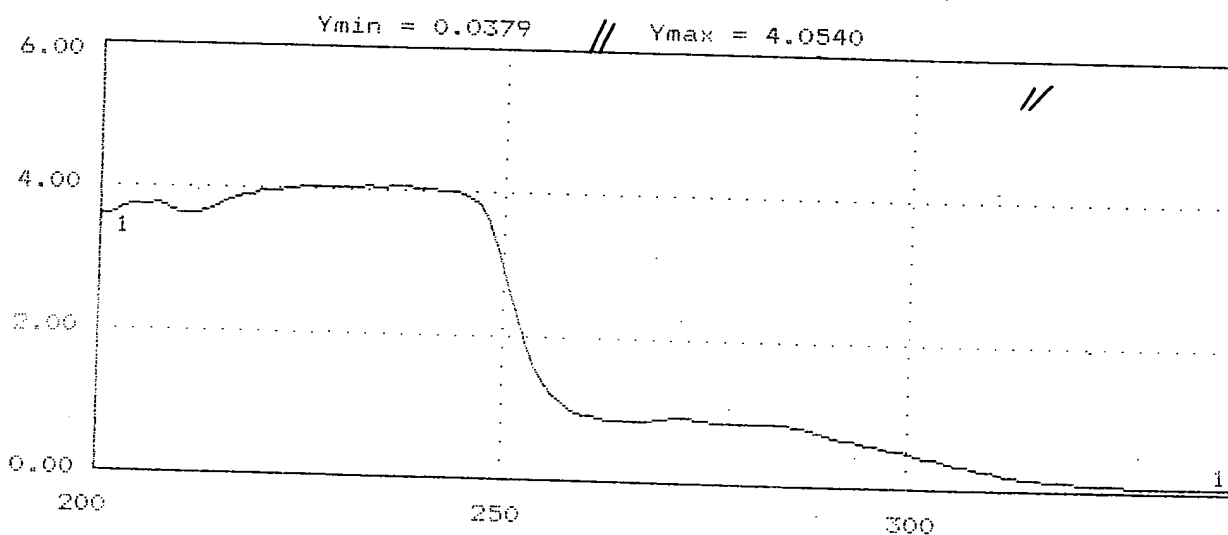
20-02.97

D Réalisation de spectres de chaque solution contre l'eau et contre le tween 80.27.

Remarque: on utilisera les solutions à 50, 100 et 150 mg/l pour déterminer la gamme étalon; la solution à 200 mg/l servira à confirmer la concentration à saturation car elle n'est toujours pas limpide après 1 nuit d'agitation et 15 minutes d'ultrasons.

SOLUTION 50MG/L A CONTRE EAU

20-02-1996 09:5



CONTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001452

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20-02-97

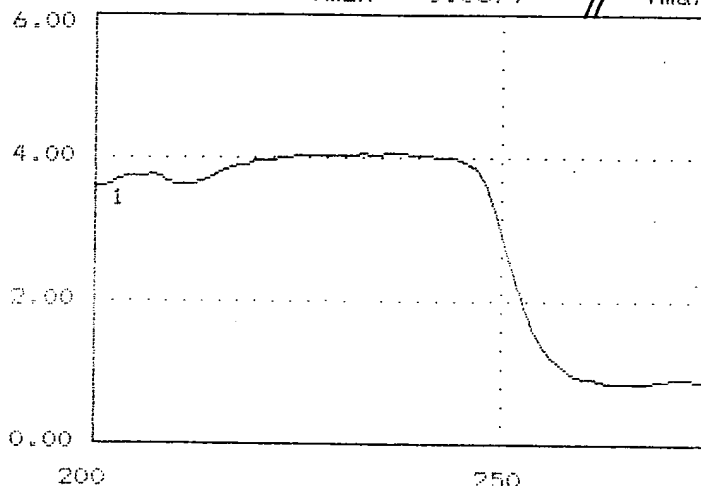
Réalisation de spectres de chaque solution contre l'eau et contre le tween 80.27.

Remarque: on utilisera les solutions à 50, 100 et 150 mg/l pour déterminer la gamme étalon; la solution à 200 mg/l servira à confirmer la concentration à saturation car elle n'est toujours pas limpide après 1 nuit d'agitation et 15 minutes d'ultrasons.

SOLUTION 50MG/L A CONTRE EAU

20-02-1996 09:58

Ymin = 0.0379 // Ymax = 4.0540



CONTRON INSTRUMENTS

50mg / A / eau

300.0	41	0.4709_1
299.0	42	0.4942_1
298.0	43	0.5153_1
297.0	44	0.5375_1
296.0	45	0.5585_1
295.0	46	0.5806_1
294.0	47	0.6012_1
293.0	48	0.6233_1
292.0	49	0.6475_1
291.0	50	0.6738_1
290.0	51	0.7001_1
289.0	52	0.7293_1
288.0	53	0.7603_1
287.0	54	0.7883_1
286.0	55	0.8129_1
285.0	56	0.8338_1
284.0	57	0.8460_1
283.0	58	0.8508_1
282.0	59	0.8476_1
281.0	60	0.8422_1
280.0	61	0.8377_1

FOURNIER 1001453

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Protective Order

5 0.11  
20-02-97

5 Réalisation de spectres de chaque solution contre l'eau et contre le tween 80.27.

Remarque: on utilisera les solutions à 50, 100 et 150 mg/l pour déterminer le gamme étalon; la solution à 200 mg/l servira à confirmer la concentration à saturation car elle n'est toujours pas limpide après 4 nuit d'agitation et 15 minutes d'ultrasons.

SOLUTION 50MG/L A CONTRE EAU //

20-02-1996 09:58

Ymin = 0.0379 //

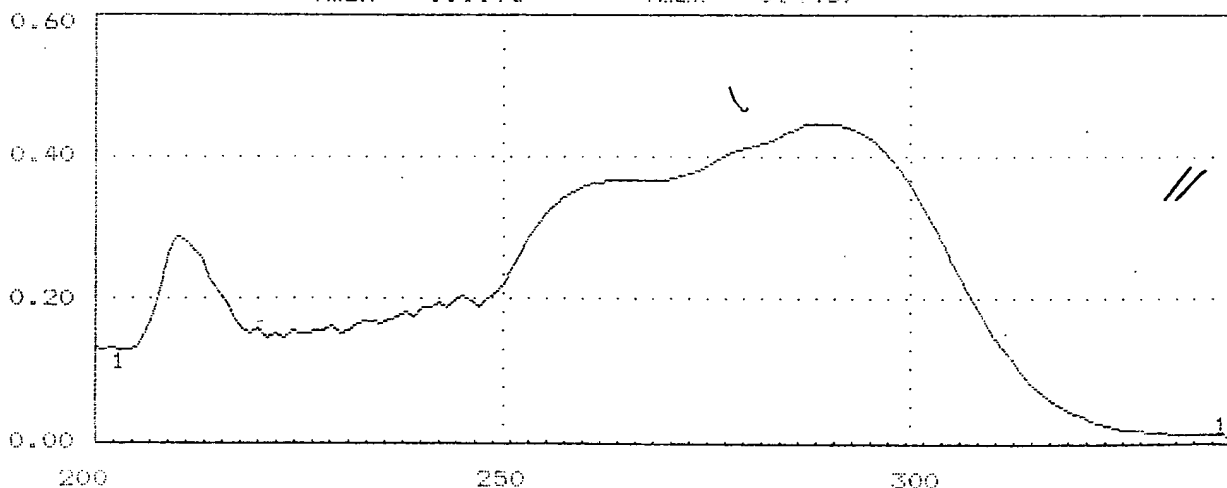
Ymax = 4.0540

SOLUTION 50MG/L A CONTRE TWEEN 80 //

20-02-1996 09:50

Ymin = 0.0095

Ymax = 0.4457 //



CONTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001454

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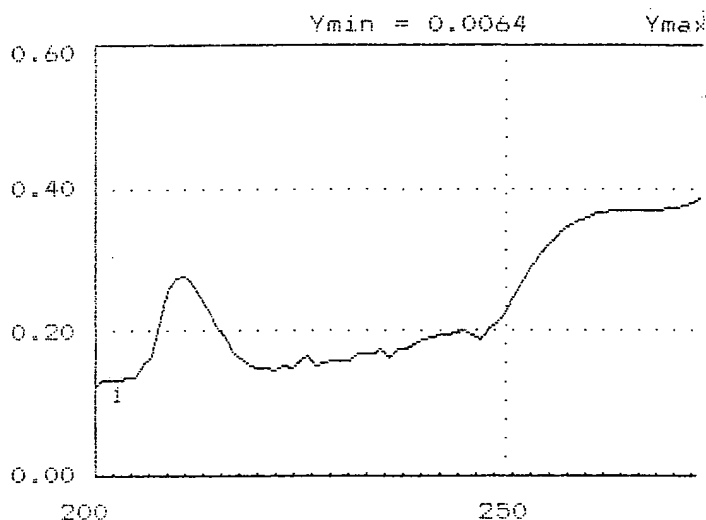
20 6/3/97

SOLUTION 50MG/L B CONTRE EAU

011

SOLUTION 50MG/L B CONTRE TWEEN 80

20-02-1996 10:13



20-02-1996 10:07

300.0	41	0.3549_1
299.0	42	0.3724_1
298.0	43	0.3875_1
297.0	44	0.4014_1
296.0	45	0.4132_1
295.0	46	0.4229_1
294.0	47	0.4288_1
293.0	48	0.4347_1
292.0	49	0.4385_1
291.0	50	0.4430_1
290.0	51	0.4432_1
289.0	52	0.4446_1
288.0	53	0.4450_1
287.0	54	0.4425_1
286.0	55	0.4381_1
285.0	56	0.4350_1
284.0	57	0.4288_1
283.0	58	0.4235_1
282.0	59	0.4189_1
281.0	60	0.4161_1
280.0	61	0.4132_1

NO B / Tween

CONTRON INSTRUMENTS

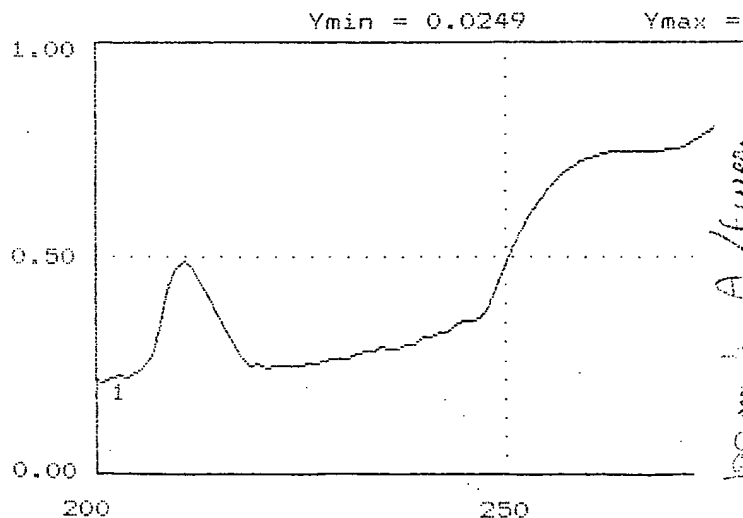
FOURNIER 1001455

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SOLUTION 100MG/L A CONTRE EAU

20-02-1996 10:21

SOLUTION 100MG/L A CONTRE TWEEN 80



20-02-1996 10:25

300.0	41	0.7104_1
299.0	42	0.7448_1
298.0	43	0.7757_1
297.0	44	0.8032_1
296.0	45	0.8267_1
295.0	46	0.8454_1
294.0	47	0.8594_1
293.0	48	0.8710_1
292.0	49	0.8796_1
291.0	50	0.8871_1
290.0	51	0.8898_1
289.0	52	0.8922_1
288.0	53	0.8932_1
287.0	54	0.8893_1
286.0	55	0.8818_1
285.0	56	0.8736_1
284.0	57	0.8635_1
283.0	58	0.8541_1
282.0	59	0.8445_1
281.0	60	0.8383_1
280.0	61	0.8335_1

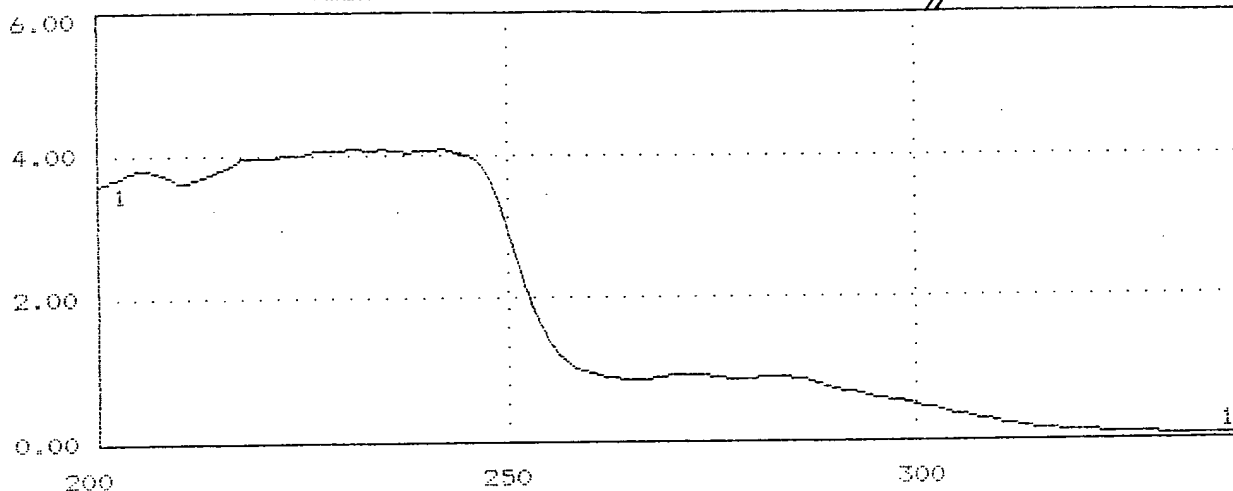
100 mg / A / Tween

CONTRON INSTRUMENTS

SOLUTION 50MG/L B CONTRE EAU

20-02-1996 10:13

Ymin = 0.0455 Ymax = 4.0780



CONTRON INSTRUMENTS

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CONTRON INSTRUMENTS

289.0	52	0.8922_1
288.0	53	0.8932_1
287.0	54	0.8893_1
286.0	55	0.8818_1
285.0	56	0.8736_1
284.0	57	0.8635_1
283.0	58	0.8541_1
282.0	59	0.8445_1
281.0	60	0.8383_1
280.0	61	0.8335_1

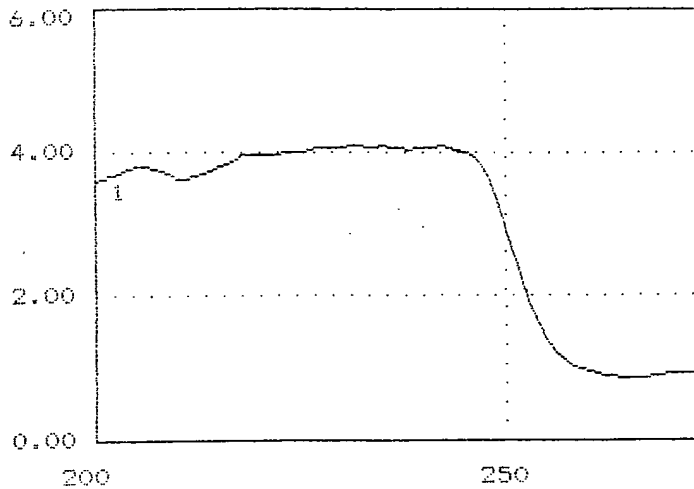
11.0

30

20-02-1996 10:13

Ymin = 0.0455

Ymax = 4.0780

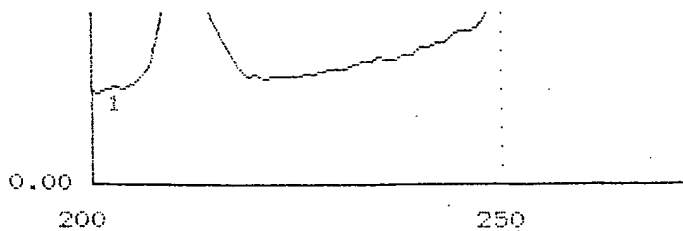


KONTRON INSTRUMENTS

50 mg B/EAU

300.0	41	0.4802_1
299.0	42	0.5036_1
298.0	43	0.5253_1
297.0	44	0.5467_1
296.0	45	0.5676_1
295.0	46	0.5896_1
294.0	47	0.6108_1
293.0	48	0.6340_1
292.0	49	0.6583_1
291.0	50	0.6839_1
290.0	51	0.7101_1
289.0	52	0.7398_1
288.0	53	0.7712_1
287.0	54	0.8001_1
286.0	55	0.8249_1
285.0	56	0.8459_1
284.0	57	0.8581_1
283.0	58	0.8635_1
282.0	59	0.8607_1
281.0	60	0.8553_1
280.0	61	0.8507_1

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KONTRON INSTRUMENTS

289.0	52	0.8922_1
288.0	53	0.8932_1
287.0	54	0.8892_1
286.0	55	0.8818_1
285.0	56	0.8736_1
284.0	57	0.8635_1
283.0	58	0.8541_1
282.0	59	0.8445_1
281.0	60	0.8383_1
280.0	61	0.8335_1

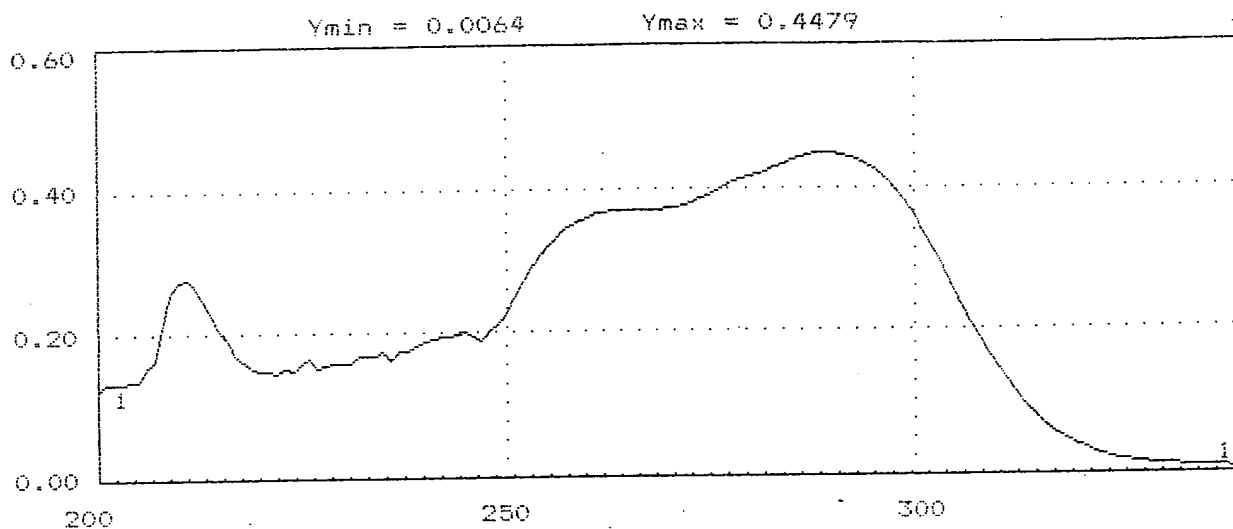
SOLUTION 50MG/L B CONTRE EAU

021

SOLUTION 50MG/L B CONTRE TWEEN 80

20-02-1996 10:13

20-02-1996 10:07



CONTRON INSTRUMENTS

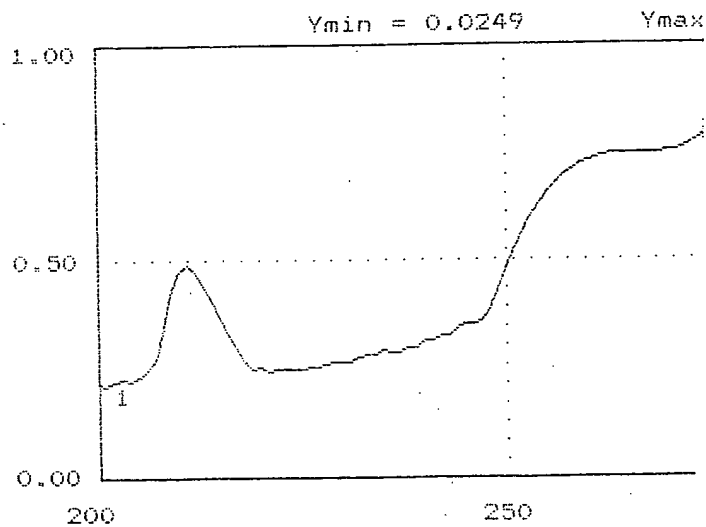
UVIKON 930

SOLUTION 100MG/L A CONTRE EAU

SOLUTION 100MG/L A CONTRE TWEEN 80

FOURNIER 1001458

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CONTRON INSTRUMENTS

100 mg/L A / Lw

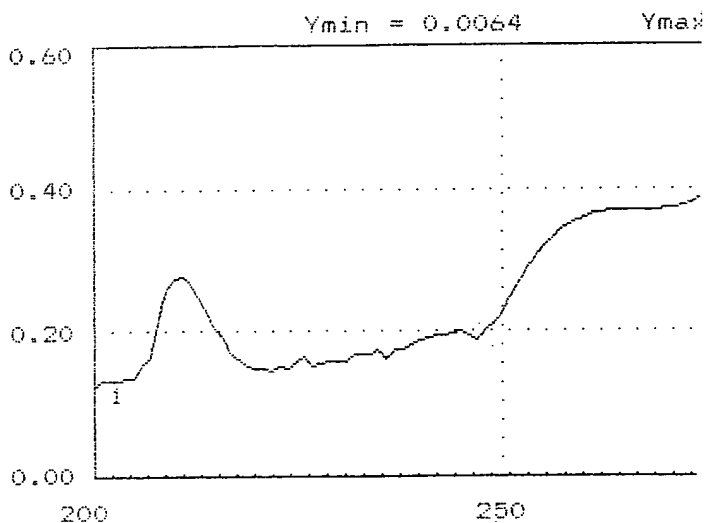
293.0	48	0.8710_1
292.0	49	0.8796_1
291.0	50	0.8871_1
290.0	51	0.8898_1
289.0	52	0.8922_1
288.0	53	0.8932_1
287.0	54	0.8893_1
286.0	55	0.8818_1
285.0	56	0.8736_1
284.0	57	0.8635_1
283.0	58	0.8541_1
282.0	59	0.8445_1
281.0	60	0.8383_1
280.0	61	0.8335_1

SOLUTION 50MG/L B CONTRE EAU

011

20-02-1996 10:13

SOLUTION 50MG/L B CONTRE TWEEN 80



20-02-1996 10:07

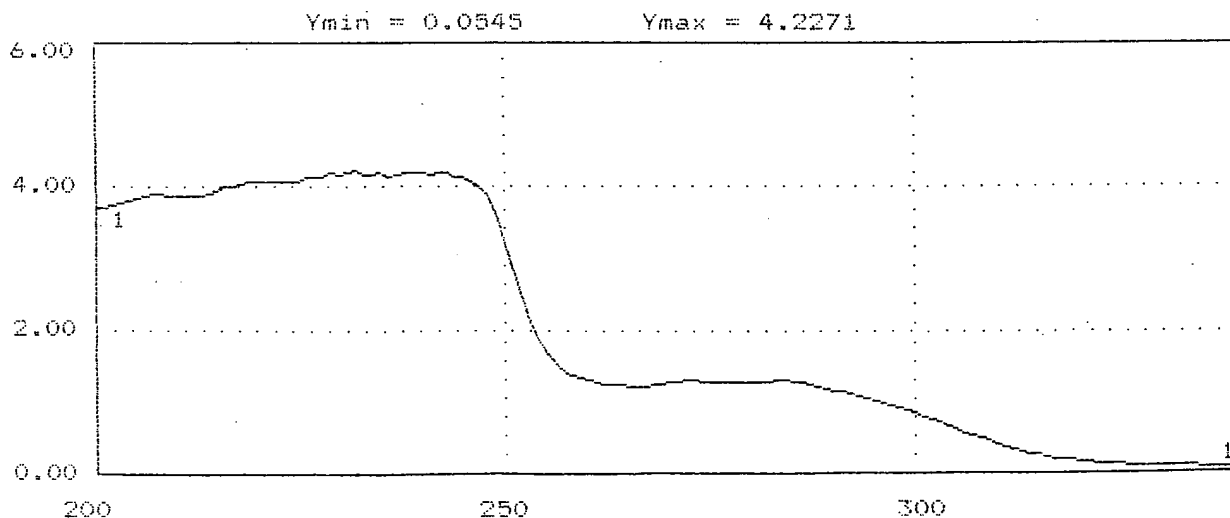
300.0	41	0.3549_1
299.0	42	0.3724_1
298.0	43	0.3875_1
297.0	44	0.4014_1
296.0	45	0.4132_1
295.0	46	0.4229_1
294.0	47	0.4288_1
293.0	48	0.4347_1
292.0	49	0.4385_1
291.0	50	0.4430_1
290.0	51	0.4432_1
289.0	52	0.4446_1
288.0	53	0.4450_1
287.0	54	0.4425_1
286.0	55	0.4381_1
285.0	56	0.4350_1
284.0	57	0.4288_1
283.0	58	0.4235_1
282.0	59	0.4189_1
281.0	60	0.4161_1
280.0	61	0.4132_1

100 Tween

KONTRON INSTRUMENTS

SOLUTION 100MG/L A CONTRE EAU

20-02-1996 10:21



KONTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001459

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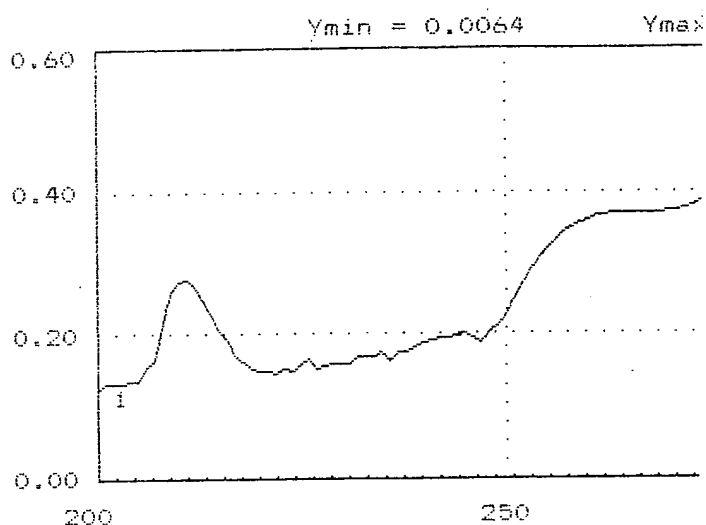
011

SOLUTION 50MG/L B CONTRE EAU

20-02-1996 10:13

SOLUTION 50MG/L B CONTRE TWEEN 80

20-02-1996 10:07



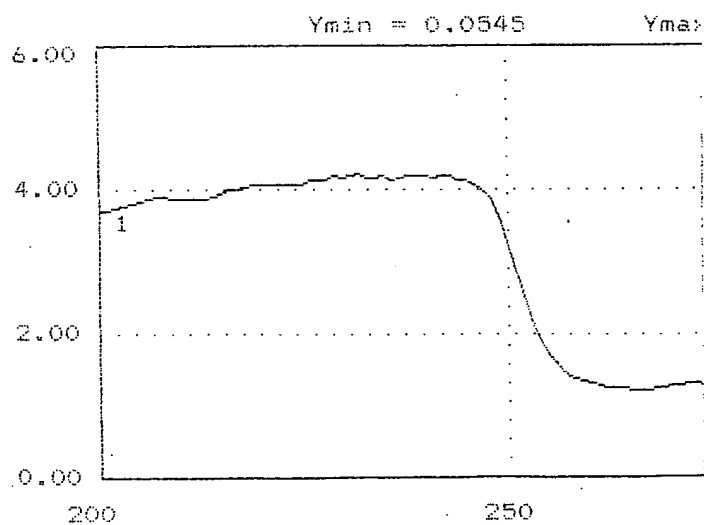
100 B / Tween

300.0	41	0.3549_1
299.0	42	0.3724_1
298.0	43	0.3875_1
297.0	44	0.4014_1
296.0	45	0.4132_1
295.0	46	0.4229_1
294.0	47	0.4288_1
293.0	48	0.4347_1
292.0	49	0.4385_1
291.0	50	0.4430_1
290.0	51	0.4432_1
289.0	52	0.4446_1
288.0	53	0.4450_1
287.0	54	0.4425_1
286.0	55	0.4381_1
285.0	56	0.4350_1
284.0	57	0.4288_1
283.0	58	0.4235_1
282.0	59	0.4189_1
281.0	60	0.4161_1
280.0	61	0.4132_1

CONTRON INSTRUMENTS

SOLUTION 100MG/L A CONTRE EAU

20-02-1996 10:21



100mg A / Eau

300.0	41	0.8236_1
299.0	42	0.8643_1
298.0	43	0.9015_1
297.0	44	0.9374_1
296.0	45	0.9697_1
295.0	46	1.0010_1
294.0	47	1.0293_1
293.0	48	1.0578_1
292.0	49	1.0865_1
291.0	50	1.1158_1
290.0	51	1.1451_1
289.0	52	1.1761_1
288.0	53	1.2081_1
287.0	54	1.2342_1
286.0	55	1.2574_1
285.0	56	1.2733_1
284.0	57	1.2815_1
283.0	58	1.2816_1
282.0	59	1.2759_1
281.0	60	1.2668_1
280.0	61	1.2587_1

CONTRON INSTRUMENTS

FOURNIER 1001460

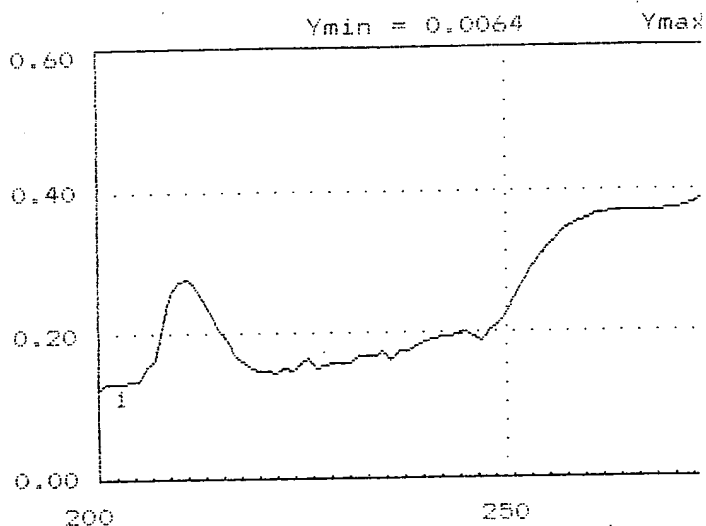
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SOLUTION 50MG/L B CONTRE EAU

20-02-1996 10:13

SOLUTION 50MG/L B CONTRE TWEEN 80

20-02-1996 10:07



50 B / Tween

300.0	41	0.3549_1
299.0	42	0.3724_1
298.0	43	0.3875_1
297.0	44	0.4014_1
296.0	45	0.4132_1
295.0	46	0.4229_1
294.0	47	0.4288_1
293.0	48	0.4347_1
292.0	49	0.4385_1
291.0	50	0.4430_1
290.0	51	0.4432_1
289.0	52	0.4446_1
288.0	53	0.4450_1
287.0	54	0.4425_1
286.0	55	0.4381_1
285.0	56	0.4350_1
284.0	57	0.4288_1
283.0	58	0.4235_1
282.0	59	0.4189_1
281.0	60	0.4161_1
280.0	61	0.4132_1

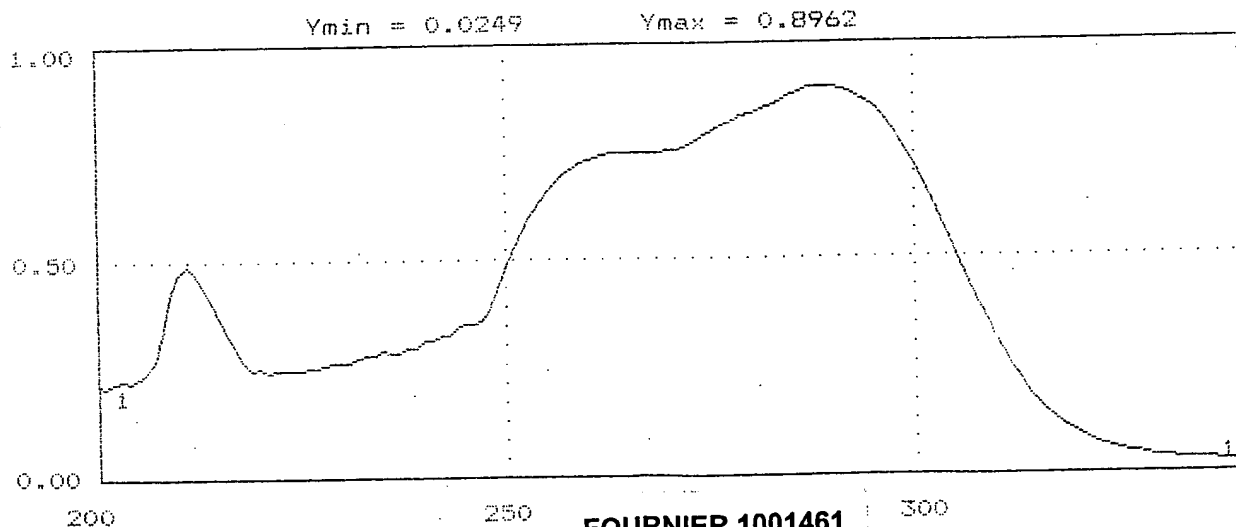
KONTRON INSTRUMENTS

SOLUTION 100MG/L A CONTRE EAU

20-02-1996 10:21

SOLUTION 100MG/L A CONTRE TWEEN 80

20-02-1996 10:25



KONTRON INSTRUMENTS

**FOURNIER 1001461**  
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Protective Order

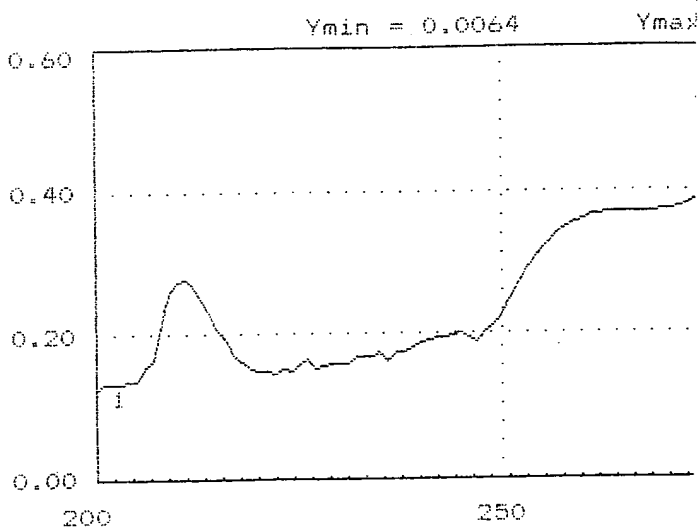
UVIKON 930

SOLUTION 50MG/L B CONTRE EAU

20-02-1996 10:13

SOLUTION 50MG/L B CONTRE TWEEN 80

20-02-1996 10:07



100 mg B / Tween

300.0	41	0.3549_1
299.0	42	0.3724_1
298.0	43	0.3875_1
297.0	44	0.4014_1
296.0	45	0.4132_1
295.0	46	0.4229_1
294.0	47	0.4288_1
293.0	48	0.4347_1
292.0	49	0.4385_1
291.0	50	0.4430_1
290.0	51	0.4432_1
289.0	52	0.4446_1
288.0	53	0.4450_1
287.0	54	0.4425_1
286.0	55	0.4381_1
285.0	56	0.4350_1
284.0	57	0.4288_1
283.0	58	0.4235_1
282.0	59	0.4189_1
281.0	60	0.4161_1
280.0	61	0.4132_1

CONTRON INSTRUMENTS

FOURNIER 1001462

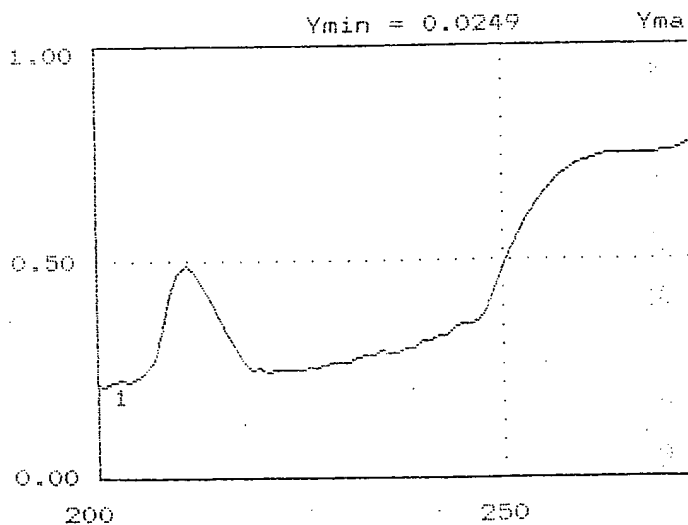
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SOLUTION 100MG/L A CONTRE EAU

20-02-1996 10:21

SOLUTION 100MG/L A CONTRE TWEEN 80

20-02-1996 10:25



100 mg A / Tween

300.0	41	0.7104_1
299.0	42	0.7448_1
298.0	43	0.7757_1
297.0	44	0.8032_1
296.0	45	0.8267_1
295.0	46	0.8454_1
294.0	47	0.8594_1
293.0	48	0.8710_1
292.0	49	0.8796_1
291.0	50	0.8871_1
290.0	51	0.8898_1
289.0	52	0.8922_1
288.0	53	0.8932_1
287.0	54	0.8893_1
286.0	55	0.8818_1
285.0	56	0.8736_1
284.0	57	0.8635_1
283.0	58	0.8541_1
282.0	59	0.8445_1
281.0	60	0.8383_1
280.0	61	0.8335_1

CONTRON INSTRUMENTS

012

SOLUTION 100MG/L B CONTRE EAU

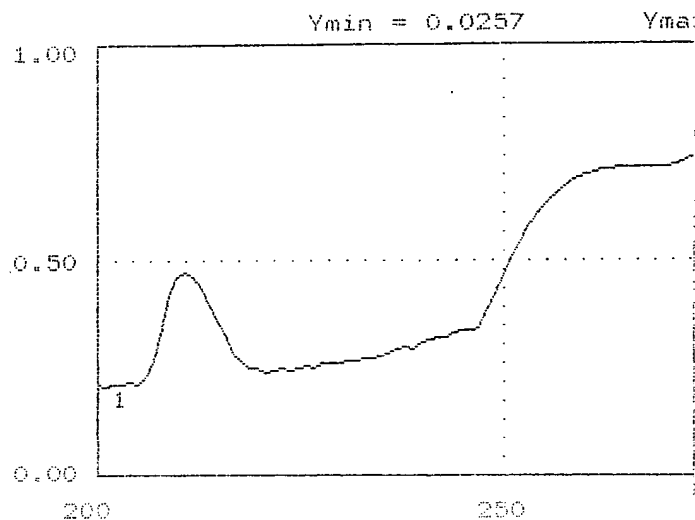
erreur

d2 collège 21/2/97

20-02-1996

10:59

SOLUTION 100MG/L B CONTRE TWEEN 80



ONTRON INSTRUMENTS

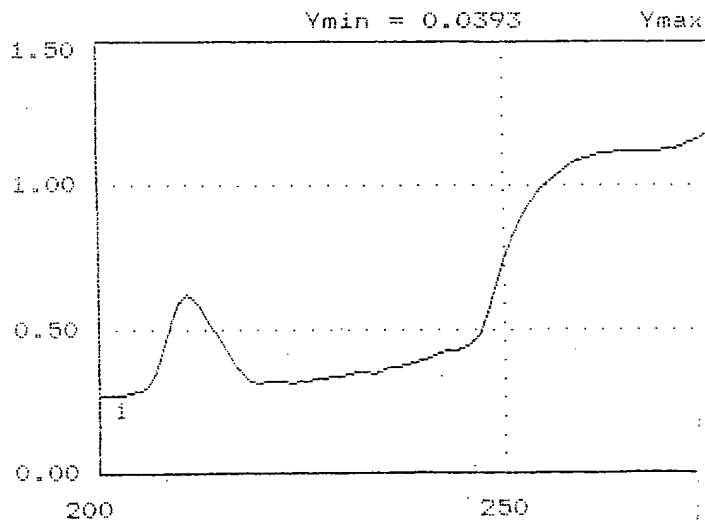
FOURNIER 1001463  
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100 / B / Tween

300.0	41	0.6789_1
299.0	42	0.7114_1
298.0	43	0.7404_1
297.0	44	0.7662_1
296.0	45	0.7882_1
295.0	46	0.8063_1
294.0	47	0.8191_1
293.0	48	0.8303_1
292.0	49	0.8388_1
291.0	50	0.8451_1
290.0	51	0.8491_1
289.0	52	0.8517_1
288.0	53	0.8527_1
287.0	54	0.8494_1
286.0	55	0.8429_1
285.0	56	0.8357_1
284.0	57	0.8265_1
283.0	58	0.8171_1
282.0	59	0.8083_1
281.0	60	0.8016_1
280.0	61	0.7966_1

SOLUTION 150MG/L A CONTRE EAU

SOLUTION 150MG/L A CONTRE TWEEN 80



ONTRON INSTRUMENTS

150 mg A / Tween

300.0	41	1.0465_1
299.0	42	1.0971_1
298.0	43	1.1448_1
297.0	44	1.1863_1
296.0	45	1.2197_1
295.0	46	1.2485_1
294.0	47	1.2692_1
293.0	48	1.2874_1
292.0	49	1.3010_1
291.0	50	1.3126_1
290.0	51	1.3192_1
289.0	52	1.3240_1
288.0	53	1.3257_1
287.0	54	1.3216_1
286.0	55	1.3120_1
285.0	56	1.3009_1
284.0	57	1.2864_1
283.0	58	1.2741_1
282.0	59	1.2607_1
281.0	60	1.2519_1
280.0	61	1.2449_1

AR 6/2/97

012

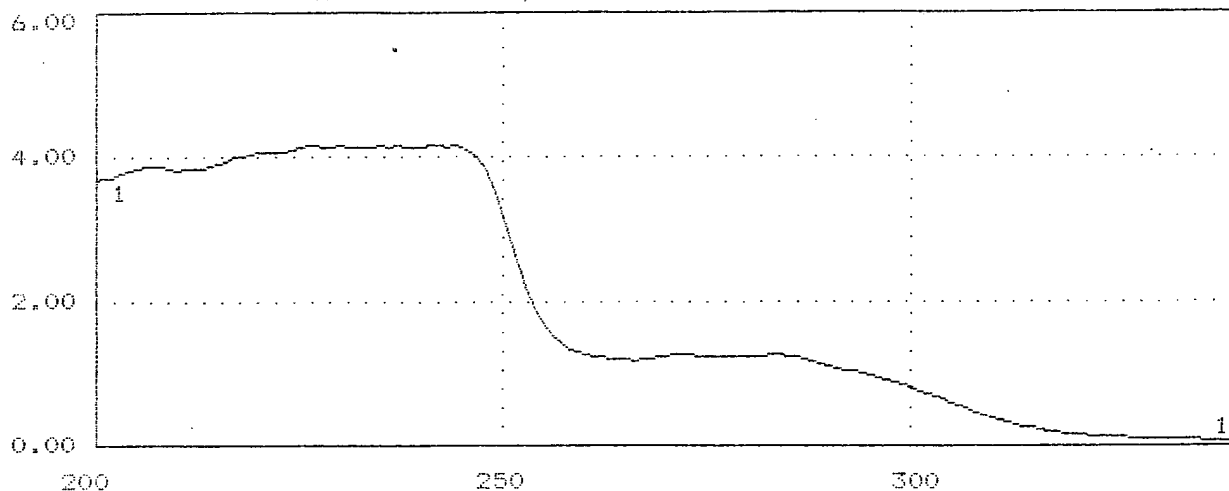
SOLUTION 100MG/L B CONTRE EAU

erreur

de collage 21/2/97

// 20/02/1996 10:59

Ymin = 0.0569 Ymax = 4.1580

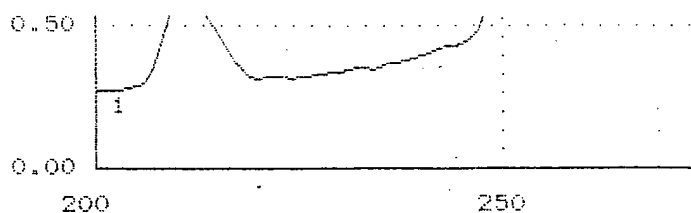


CONTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001464

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CONTRON INSTRUMENTS

288.0	53	1.3257_1
287.0	54	1.3216_1
286.0	55	1.3120_1
285.0	56	1.3009_1
284.0	57	1.2864_1
283.0	58	1.2741_1
282.0	59	1.2607_1
281.0	60	1.2519_1
280.0	61	1.2449_1

AP. 6/2/97

012

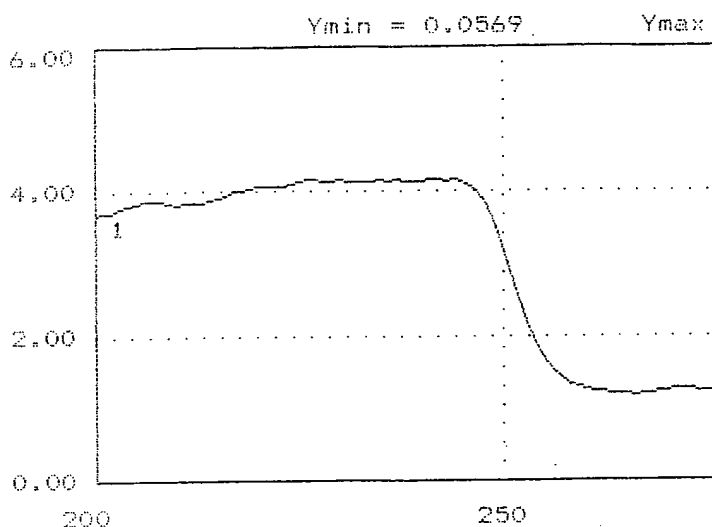
SOLUTION 100MB/L B CONTRE EAU

erreur

de collège 21/2/97

CC

20/02/1996 10:59



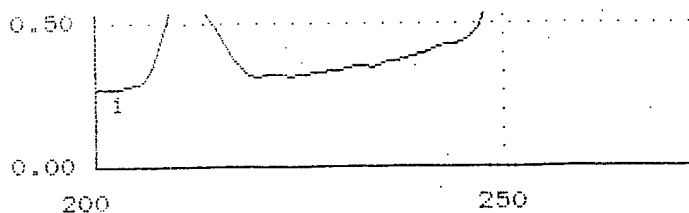
100 B / eau

300.0	41	0.7924_1
299.0	42	0.8311_1
298.0	43	0.8668_1
297.0	44	0.9017_1
296.0	45	0.9335_1
295.0	46	0.9641_1
294.0	47	0.9923_1
293.0	48	1.0199_1
292.0	49	1.0483_1
291.0	50	1.0782_1
290.0	51	1.1069_1
289.0	52	1.1381_1
288.0	53	1.1690_1
287.0	54	1.1972_1
286.0	55	1.2217_1
285.0	56	1.2390_1
284.0	57	1.2481_1
283.0	58	1.2492_1
282.0	59	1.2430_1
281.0	60	1.2345_1
280.0	61	1.2273_1

CONTRON INSTRUMENTS

FOURNIER 1001465

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288.0	53	1.3257_1
287.0	54	1.3216_1
286.0	55	1.3120_1
285.0	56	1.3009_1
284.0	57	1.2864_1
283.0	58	1.2741_1
282.0	59	1.2607_1
281.0	60	1.2519_1
280.0	61	1.2449_1

CONTRON INSTRUMENTS

20.02/97

012

SOLUTION 100MG/L B CONTRE EAU

erreur

de collage 21/2/97

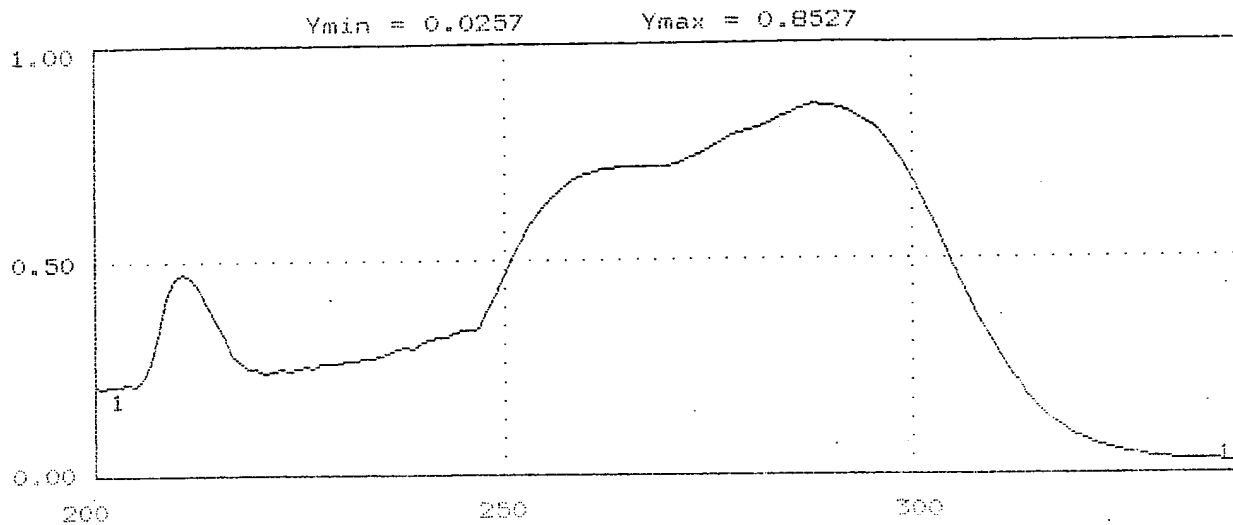
20-02-1996

10:59

SOLUTION 100MG/L B CONTRE TWEEN 80

20-02-1996

10:45



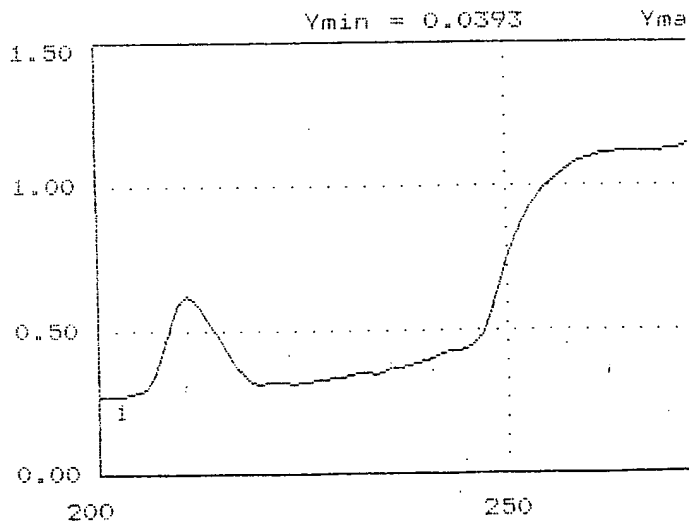
CONTRON INSTRUMENTS

UVIKON 930

SOLUTION 150MG/L A CONTRE EAU

SOLUTION 150MG/L A CONTRE TWEEN 80

FOURNIER 1001466

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Protective Order

ASD Ang A/Lu

293.0	48	1.2874_1
292.0	49	1.3010_1
291.0	50	1.3126_1
290.0	51	1.3192_1
289.0	52	1.3240_1
288.0	53	1.3257_1
287.0	54	1.3216_1
286.0	55	1.3120_1
285.0	56	1.3009_1
284.0	57	1.2864_1
283.0	58	1.2741_1
282.0	59	1.2607_1
281.0	60	1.2519_1
280.0	61	1.2449_1

CONTRON INSTRUMENTS

de collage

012

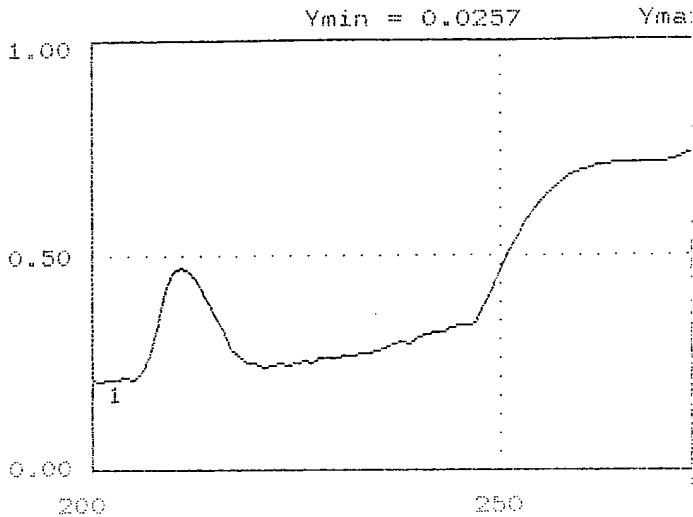
SOLUTION 100MG/L B CONTRE EAU

erreur

de collage 2/2/96

20-02-1996 10:59

SOLUTION 100MG/L B CONTRE TWEEN 80



CONTRON INSTRUMENTS

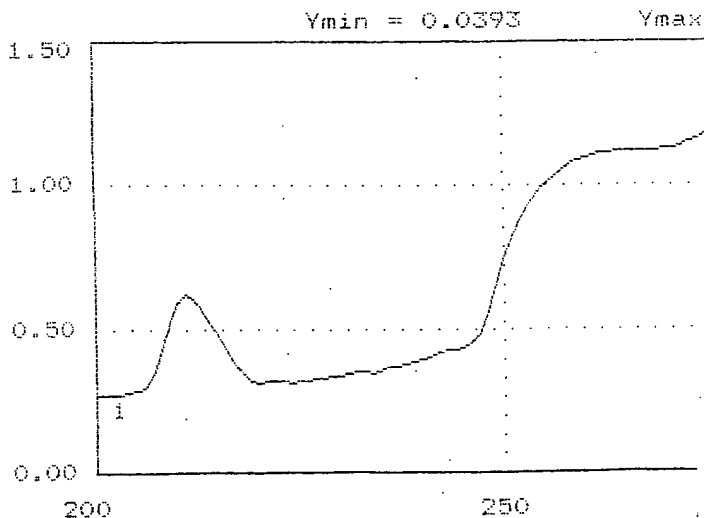
FOURNIER 1001467

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Protective Order

SOLUTION 150MG/L A CONTRE EAU

20-02-1996 11:03

SOLUTION 150MG/L A CONTRE TWEEN 80



CONTRON INSTRUMENTS

de collage

20-02-1996 10:45

100 / B / Tween

300.0	41	0.6789_1
299.0	42	0.7114_1
298.0	43	0.7404_1
297.0	44	0.7662_1
296.0	45	0.7882_1
295.0	46	0.8063_1
294.0	47	0.8191_1
293.0	48	0.8303_1
292.0	49	0.8388_1
291.0	50	0.8451_1
290.0	51	0.8491_1
289.0	52	0.8517_1
288.0	53	0.8527_1
287.0	54	0.8494_1
286.0	55	0.8429_1
285.0	56	0.8357_1
284.0	57	0.8265_1
283.0	58	0.8171_1
282.0	59	0.8083_1
281.0	60	0.8016_1
280.0	61	0.7966_1

20-02-1996 11:10

150 mg A / Tween

300.0	41	1.0465_1
299.0	42	1.0971_1
298.0	43	1.1448_1
297.0	44	1.1863_1
296.0	45	1.2197_1
295.0	46	1.2485_1
294.0	47	1.2692_1
293.0	48	1.2874_1
292.0	49	1.3010_1
291.0	50	1.3126_1
290.0	51	1.3192_1
289.0	52	1.3240_1
288.0	53	1.3257_1
287.0	54	1.3216_1
286.0	55	1.3120_1
285.0	56	1.3009_1
284.0	57	1.2864_1
283.0	58	1.2741_1
282.0	59	1.2607_1
281.0	60	1.2519_1
280.0	61	1.2449_1



012

SOLUTION 100MG/L B CONTRE EAU

erreur

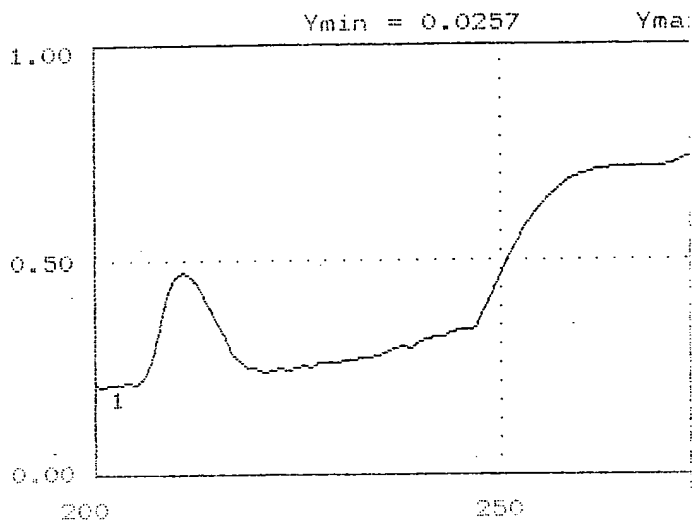
de collage 2/12/96

CC

20-02-1996

10:59

SOLUTION 100MG/L B CONTRE TWEEN 80



100 / B / Tween

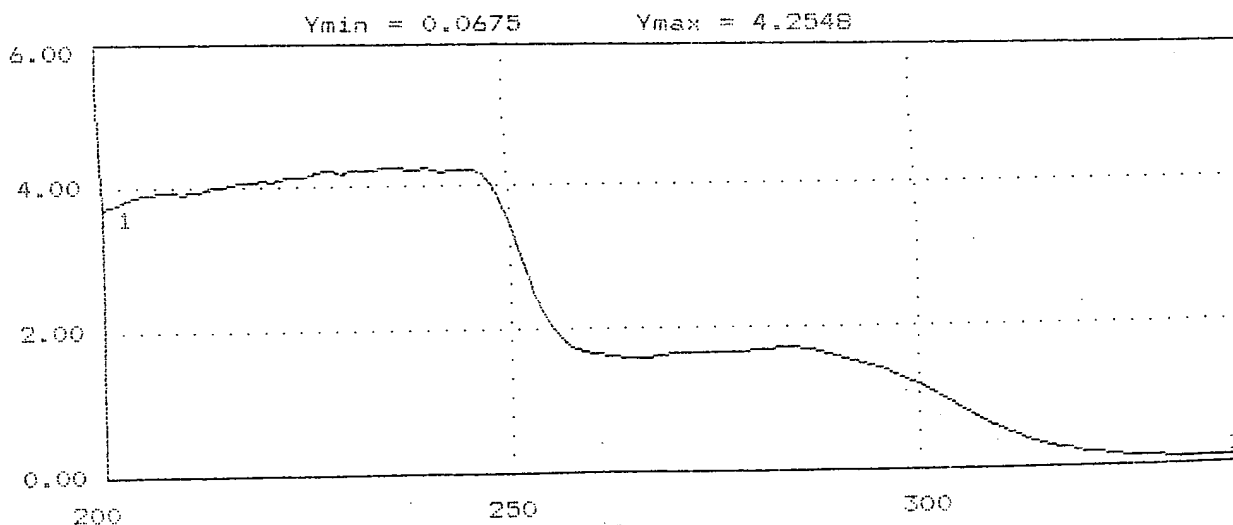
300.0	41	0.6789_1
299.0	42	0.7114_1
298.0	43	0.7404_1
297.0	44	0.7662_1
296.0	45	0.7882_1
295.0	46	0.8063_1
294.0	47	0.8191_1
293.0	48	0.8303_1
292.0	49	0.8388_1
291.0	50	0.8451_1
290.0	51	0.8491_1
289.0	52	0.8517_1
288.0	53	0.8527_1
287.0	54	0.8494_1
286.0	55	0.8429_1
285.0	56	0.8357_1
284.0	57	0.8265_1
283.0	58	0.8171_1
282.0	59	0.8083_1
281.0	60	0.8016_1
280.0	61	0.7966_1

CONTRON INSTRUMENTS

SOLUTION 150MG/L A CONTRE EAU

20-02-1996

11:03



CONTRON INSTRUMENTS

UVIKON 93

FOURNIER 1001468

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012

SOLUTION 100MG/L B CONTRE EAU

erreur

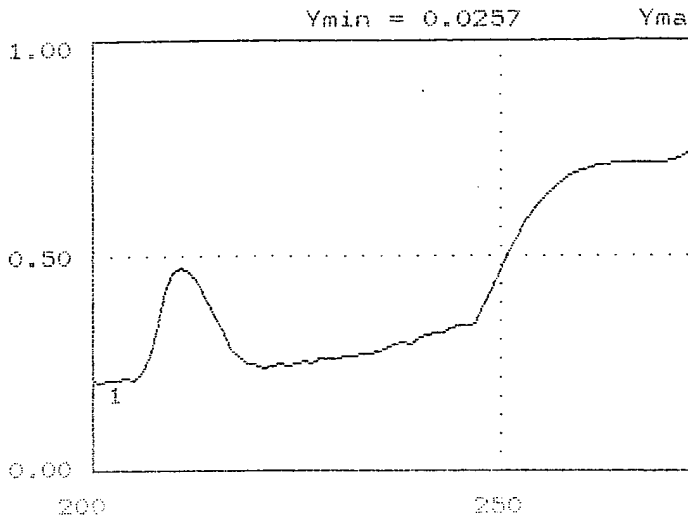
de collage 21/2/97

CC

20-02-1996

10:59

SOLUTION 100MG/L B CONTRE TWEEN 80



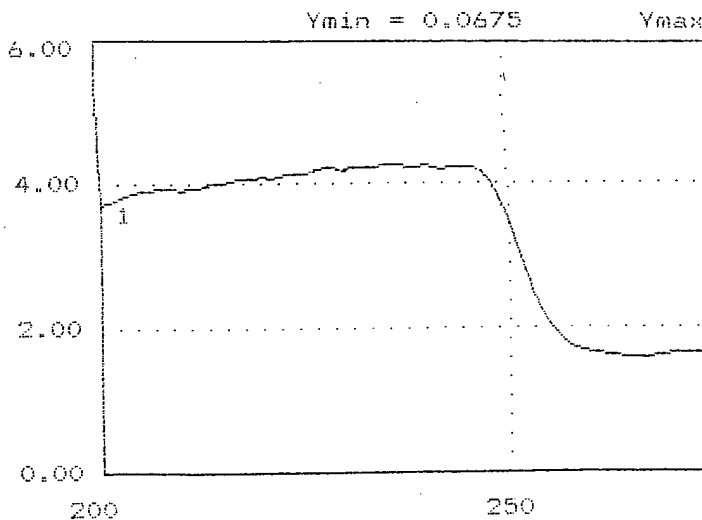
CONTRON INSTRUMENTS

100 / B / Tween

20-02-1996 10:45

300.0	41	0.6789_1
299.0	42	0.7114_1
298.0	43	0.7404_1
297.0	44	0.7662_1
296.0	45	0.7882_1
295.0	46	0.8063_1
294.0	47	0.8191_1
293.0	48	0.8303_1
292.0	49	0.8388_1
291.0	50	0.8451_1
290.0	51	0.8491_1
289.0	52	0.8517_1
288.0	53	0.8527_1
287.0	54	0.8494_1
286.0	55	0.8429_1
285.0	56	0.8357_1
284.0	57	0.8265_1
283.0	58	0.8171_1
282.0	59	0.8083_1
281.0	60	0.8016_1
280.0	61	0.7966_1

SOLUTION 150MG/L A CONTRE EAU



CONTRON INSTRUMENTS

150 A / Eau

20-02-1996 11:07

300.0	41	1.1486_1
299.0	42	1.2044_1
298.0	43	1.2560_1
297.0	44	1.3049_1
296.0	45	1.3459_1
295.0	46	1.3874_1
294.0	47	1.4242_1
293.0	48	1.4578_1
292.0	49	1.4899_1
291.0	50	1.5240_1
290.0	51	1.5537_1
289.0	52	1.5861_1
288.0	53	1.6183_1
287.0	54	1.6452_1
286.0	55	1.6660_1
285.0	56	1.6788_1
284.0	57	1.6823_1
283.0	58	1.6795_1
282.0	59	1.6680_1
281.0	60	1.6556_1
280.0	61	1.6487_1

FOURNIER 1001469

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Protective Order

012

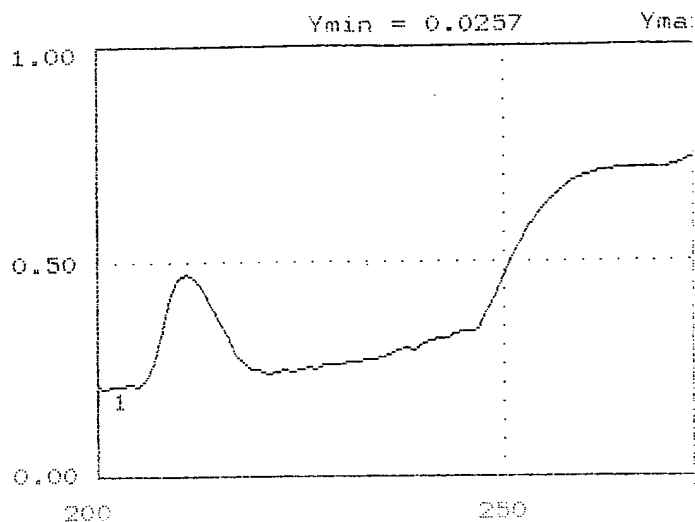
SOLUTION 100MG/L B CONTRE EAU

erreur

de collage 21/2/97

20-02-1996 10:59

SOLUTION 100MG/L B CONTRE TWEEN 80



20-02-1996 10:45

100 / B / Tween

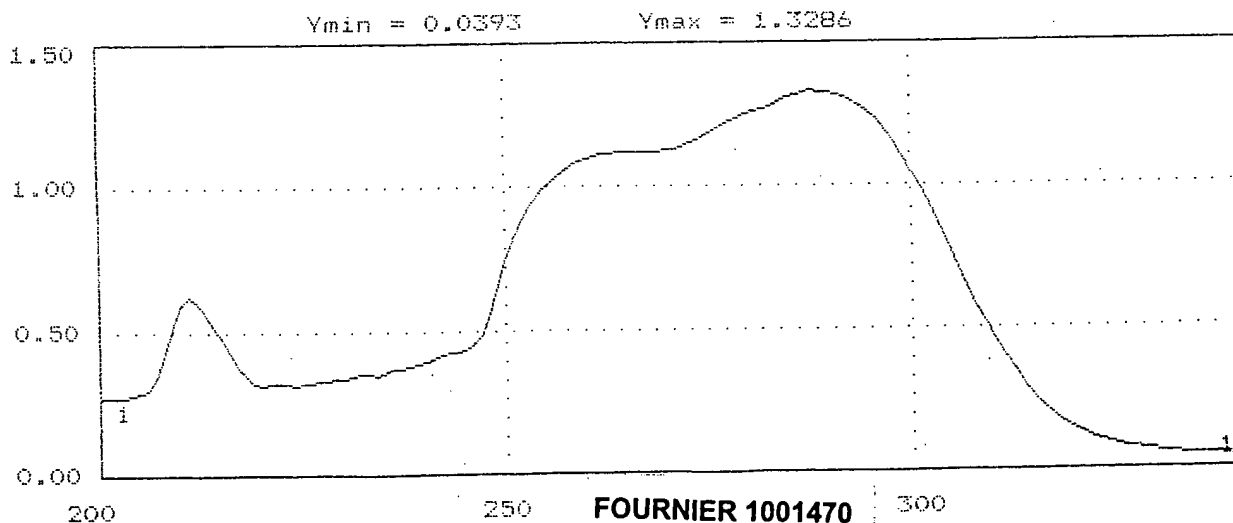
300.0	41	0.6789_1
299.0	42	0.7114_1
298.0	43	0.7404_1
297.0	44	0.7662_1
296.0	45	0.7982_1
295.0	46	0.8063_1
294.0	47	0.8191_1
293.0	48	0.8303_1
292.0	49	0.8388_1
291.0	50	0.8451_1
290.0	51	0.8491_1
289.0	52	0.8517_1
288.0	53	0.8527_1
287.0	54	0.8494_1
286.0	55	0.8429_1
285.0	56	0.8357_1
284.0	57	0.8265_1
283.0	58	0.8171_1
282.0	59	0.8083_1
281.0	60	0.8016_1
280.0	61	0.7966_1

CONTRON INSTRUMENTS

SOLUTION 150MG/L A CONTRE EAU

SOLUTION 150MG/L A CONTRE TWEEN 80

20-02-1996 11:07



CONTRON INSTRUMENTS

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UVIKON 930

20-02-1997

017

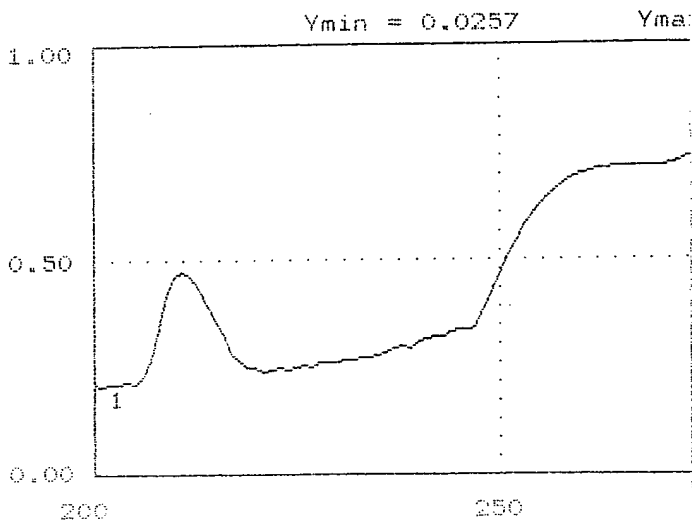
SOLUTION 100MG/L B CONTRE EAU

erreur

de collage 21/2/97

20-02-1996 10:59

SOLUTION 100MG/L B CONTRE TWEEN 80



CONTRON INSTRUMENTS

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Protective Order

FOURNIER 1001471

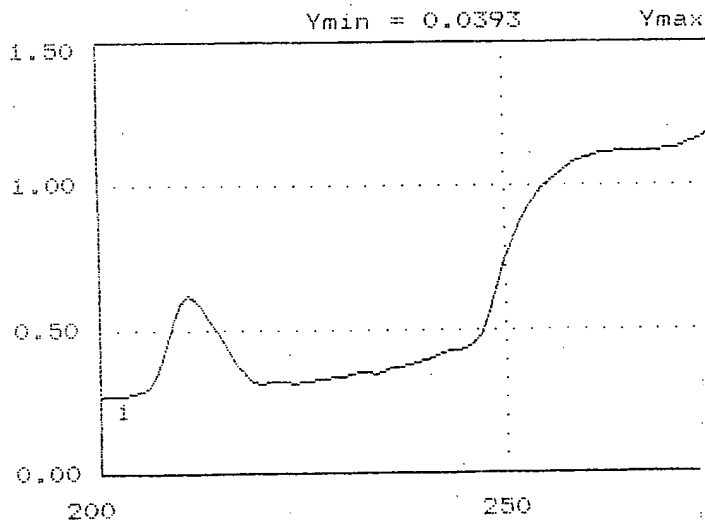
100 / B / Tween

300.0	41	0.6789_1
299.0	42	0.7114_1
298.0	43	0.7404_1
297.0	44	0.7662_1
296.0	45	0.7882_1
295.0	46	0.8063_1
294.0	47	0.8191_1
293.0	48	0.8303_1
292.0	49	0.8388_1
291.0	50	0.8451_1
290.0	51	0.8491_1
289.0	52	0.8517_1
288.0	53	0.8527_1
287.0	54	0.8494_1
286.0	55	0.8429_1
285.0	56	0.8357_1
284.0	57	0.8265_1
283.0	58	0.8171_1
282.0	59	0.8083_1
281.0	60	0.8016_1
280.0	61	0.7966_1

SOLUTION 150MG/L A CONTRE EAU

SOLUTION 150MG/L A CONTRE TWEEN 80

20-02-1996 11:07



CONTRON INSTRUMENTS

150mg A / Tween

300.0	41	1.0465_1
299.0	42	1.0971_1
298.0	43	1.1448_1
297.0	44	1.1863_1
296.0	45	1.2197_1
295.0	46	1.2485_1
294.0	47	1.2692_1
293.0	48	1.2874_1
292.0	49	1.3010_1
291.0	50	1.3126_1
290.0	51	1.3192_1
289.0	52	1.3240_1
288.0	53	1.3257_1
287.0	54	1.3216_1
286.0	55	1.3120_1
285.0	56	1.3009_1
284.0	57	1.2864_1
283.0	58	1.2741_1
282.0	59	1.2607_1
281.0	60	1.2519_1
280.0	61	1.2449_1

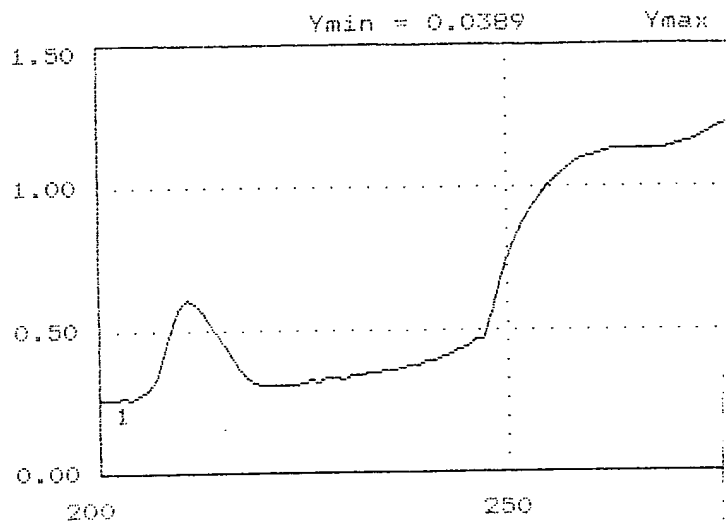
AP. C / 2/97

013

SOLUTION 150MG/L B CONTRE EAU

20-02-1996 11:25

SOLUTION 150MG/L B CONTRE TWEEN 80



20-02-1996 11:19

300.0	41	1.0579_1
299.0	42	1.1100_1
298.0	43	1.1563_1
297.0	44	1.1974_1
296.0	45	1.2327_1
295.0	46	1.2612_1
294.0	47	1.2825_1
293.0	48	1.3002_1
292.0	49	1.3131_1
291.0	50	1.3255_1
290.0	51	1.3311_1
289.0	52	1.3372_1
288.0	53	1.3381_1
287.0	54	1.3334_1
286.0	55	1.3242_1
285.0	56	1.3136_1
284.0	57	1.2981_1
283.0	58	1.2842_1
282.0	59	1.2713_1
281.0	60	1.2616_1
280.0	61	1.2555_1

150 B / Tween

CONTRON INSTRUMENTS

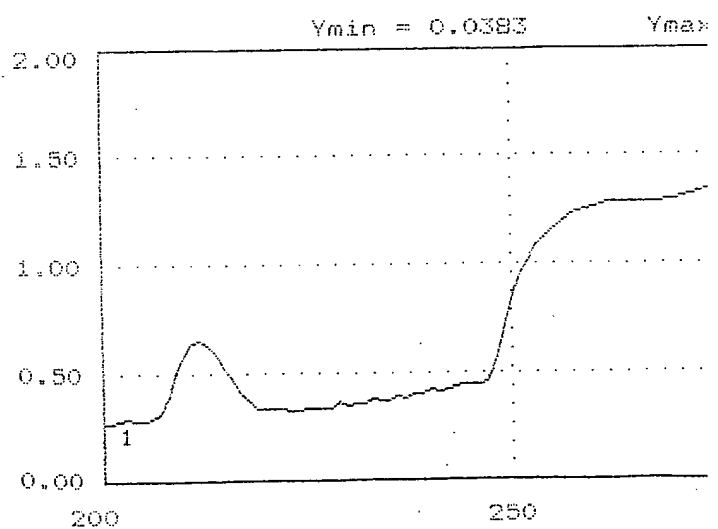
FOURNIER 1001472

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Subject to  
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SOLUTION 200MG/L A CONTRE EAU

20-02-1996 11:31

SOLUTION 200MG/L A CONTRE TWEEN 80



20-02-1996 13:31

300.0	41	1.1792_1
299.0	42	1.2388_1
298.0	43	1.2925_1
297.0	44	1.3409_1
296.0	45	1.3827_1
295.0	46	1.4182_1
294.0	47	1.4447_1
293.0	48	1.4660_1
292.0	49	1.4816_1
291.0	50	1.4961_1
290.0	51	1.5044_1
289.0	52	1.5120_1
288.0	53	1.5144_1
287.0	54	1.5115_1
286.0	55	1.5031_1
285.0	56	1.4916_1
284.0	57	1.4764_1
283.0	58	1.4615_1
282.0	59	1.4465_1
281.0	60	1.4347_1
280.0	61	1.4318_1

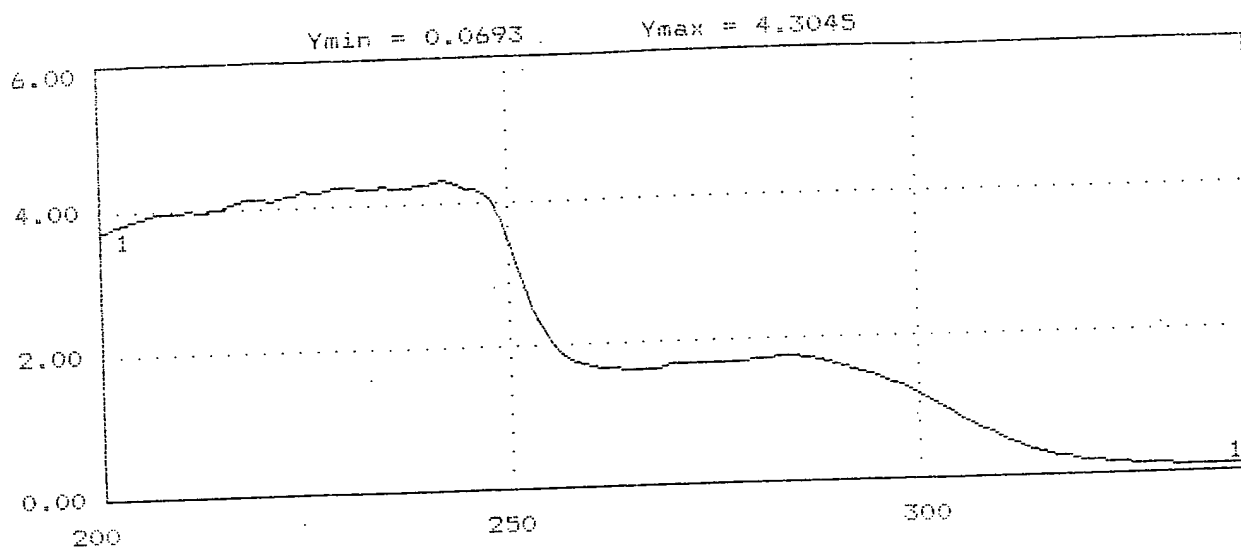
200 A / Tween 80

CONTRON INSTRUMENTS

013

SOLUTION 150MG/L B CONTRE EAU

// 20-02-1996 11:25

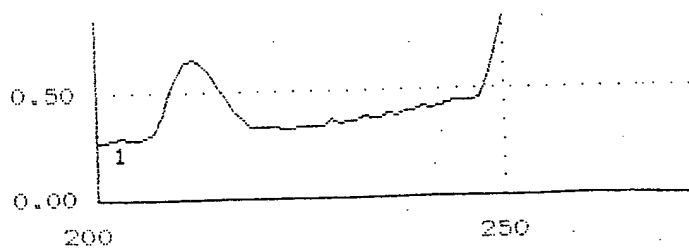


CONTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001473

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Protective Order

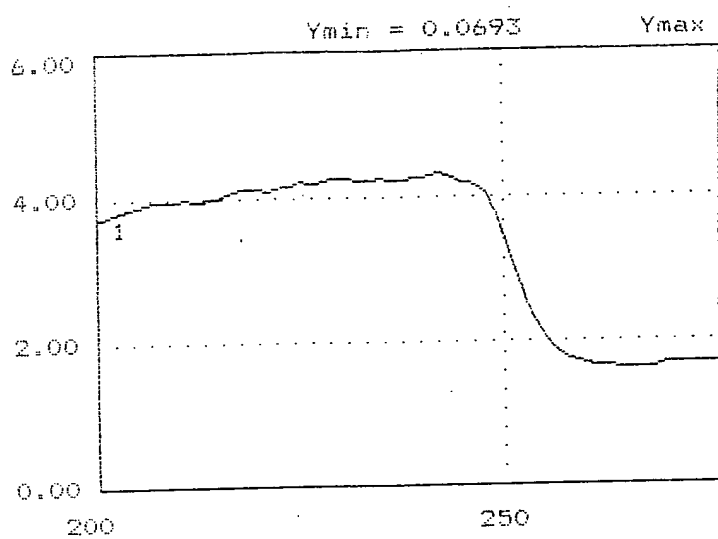


200 A / 30

287.0	52	1.5120_1
288.0	53	1.5144_1
287.0	54	1.5115_1
286.0	55	1.5031_1
285.0	56	1.4916_1
284.0	57	1.4764_1
283.0	58	1.4615_1
282.0	59	1.4465_1
281.0	60	1.4347_1
280.0	61	1.4318_1

CONTRON INSTRUMENTS

SOLUTION 150MG/L B CONTRE EAU



20-02-1996 11:25

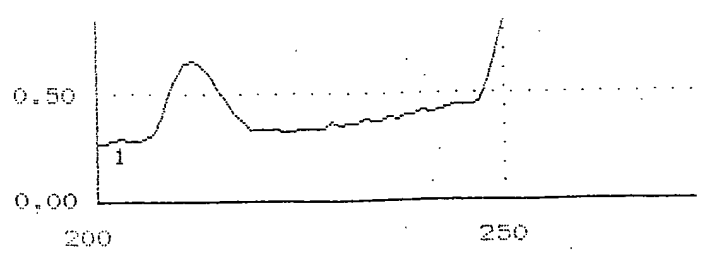
150 B/ear

300.0	41	1.1686_1
299.0	42	1.2265_1
298.0	43	1.2786_1
297.0	44	1.3283_1
296.0	45	1.3717_1
295.0	46	1.4136_1
294.0	47	1.4506_1
293.0	48	1.4851_1
292.0	49	1.5184_1
291.0	50	1.5519_1
290.0	51	1.5845_1
289.0	52	1.6170_1
288.0	53	1.6491_1
287.0	54	1.6767_1
286.0	55	1.6962_1
285.0	56	1.7089_1
284.0	57	1.7122_1
283.0	58	1.7088_1
282.0	59	1.6984_1
281.0	60	1.6871_1
280.0	61	1.6796_1

CONTRON INSTRUMENTS

FOURNIER 1001474

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Protective Order



200 A/3

287.0	52	1.5120_1
288.0	53	1.5144_1
287.0	54	1.5115_1
286.0	55	1.5031_1
285.0	56	1.4916_1
284.0	57	1.4764_1
283.0	58	1.4615_1
282.0	59	1.4465_1
281.0	60	1.4347_1
280.0	61	1.4318_1

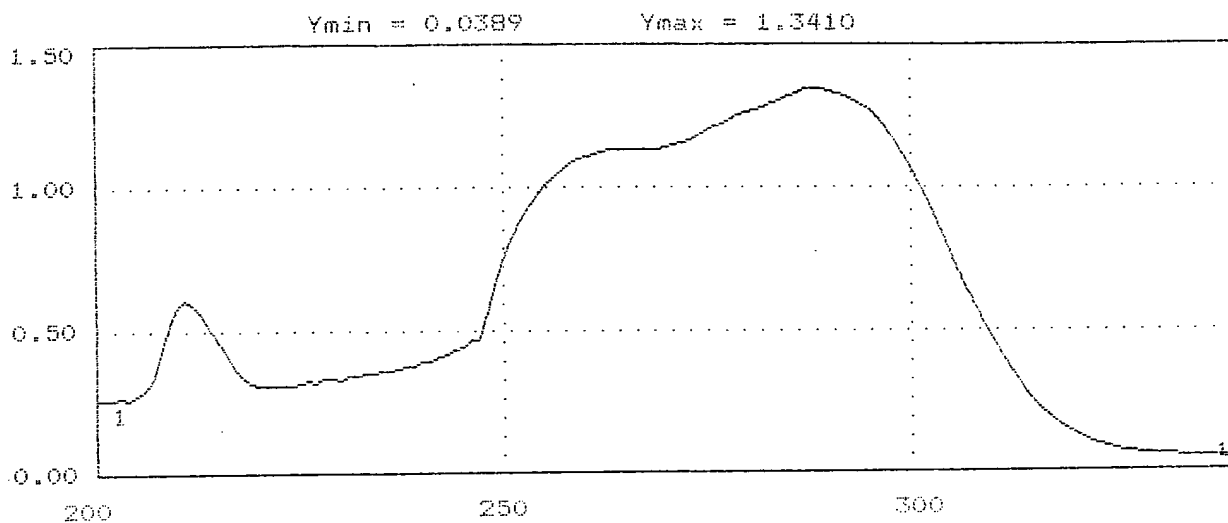
CONTRON INSTRUMENTS

SOLUTION 150MG/L B CONTRE EAU

20-02-1996 11:25

SOLUTION 150MG/L B CONTRE TWEEN 80

20-02-1996 11:19



CONTRON INSTRUMENTS

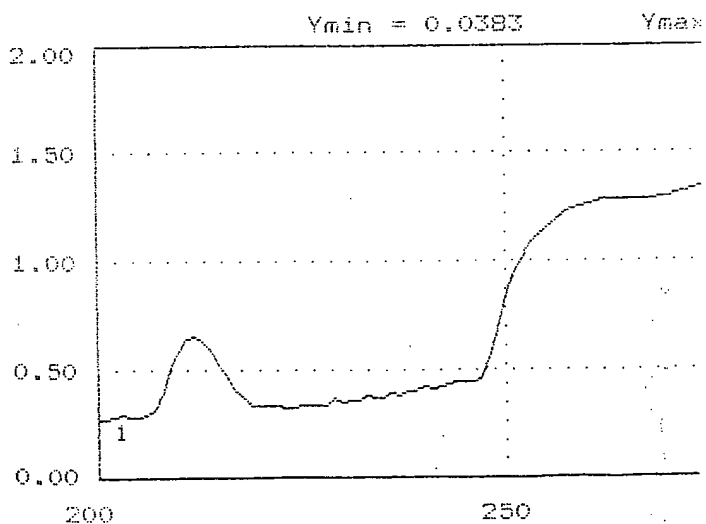
UVIKON 930

SOLUTION 200MG/L A CONTRE EAU

FOURNIER 1001475

Highly Confidential  
Subject to  
Protective Order

SOLUTION 200MG/L A CONTRE TWEEN 80



CONTRON INSTRUMENTS

*200 A / 200 mg*

293.0	48	1.4660_1
292.0	49	1.4816_1
291.0	50	1.4961_1
290.0	51	1.5044_1
289.0	52	1.5120_1
288.0	53	1.5144_1
287.0	54	1.5115_1
286.0	55	1.5031_1
285.0	56	1.4916_1
284.0	57	1.4764_1
283.0	58	1.4615_1
282.0	59	1.4465_1
281.0	60	1.4347_1
280.0	61	1.4318_1

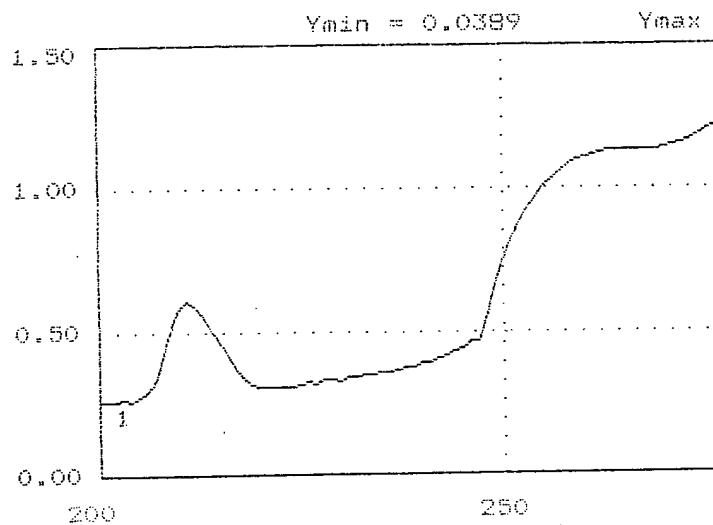


0137

SOLUTION 150MG/L B CONTRE EAU

20-02-1996 11:25

SOLUTION 150MG/L B CONTRE TWEEN 80.



CONTROIN INSTRUMENTS

FOURNIER 1001476

Highly Confidential  
Subject to  
Protective Order

20-02-1996 11:19

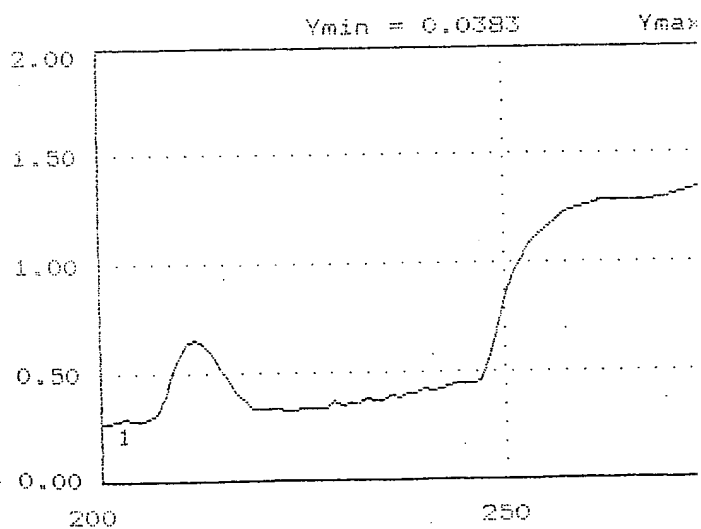
300.0	41	1.0579_1
299.0	42	1.1100_1
298.0	43	1.1563_1
297.0	44	1.1974_1
296.0	45	1.2327_1
295.0	46	1.2612_1
294.0	47	1.2825_1
293.0	48	1.3002_1
292.0	49	1.3131_1
291.0	50	1.3255_1
290.0	51	1.3311_1
289.0	52	1.3372_1
288.0	53	1.3381_1
287.0	54	1.3334_1
286.0	55	1.3242_1
285.0	56	1.3136_1
284.0	57	1.2981_1
283.0	58	1.2842_1
282.0	59	1.2713_1
281.0	60	1.2616_1
280.0	61	1.2555_1

150 B / Tween

SOLUTION 200MG/L A CONTRE EAU

20-02-1996 11:31

SOLUTION 200MG/L A CONTRE TWEEN 80



CONTROIN INSTRUMENTS

20-02-1996 13:31

300.0	41	1.1792_1
299.0	42	1.2388_1
298.0	43	1.2925_1
297.0	44	1.3409_1
296.0	45	1.3827_1
295.0	46	1.4182_1
294.0	47	1.4447_1
293.0	48	1.4660_1
292.0	49	1.4816_1
291.0	50	1.4961_1
290.0	51	1.5044_1
289.0	52	1.5120_1
288.0	53	1.5144_1
287.0	54	1.5115_1
286.0	55	1.5031_1
285.0	56	1.4916_1
284.0	57	1.4764_1
283.0	58	1.4615_1
282.0	59	1.4465_1
281.0	60	1.4347_1
280.0	61	1.4318_1

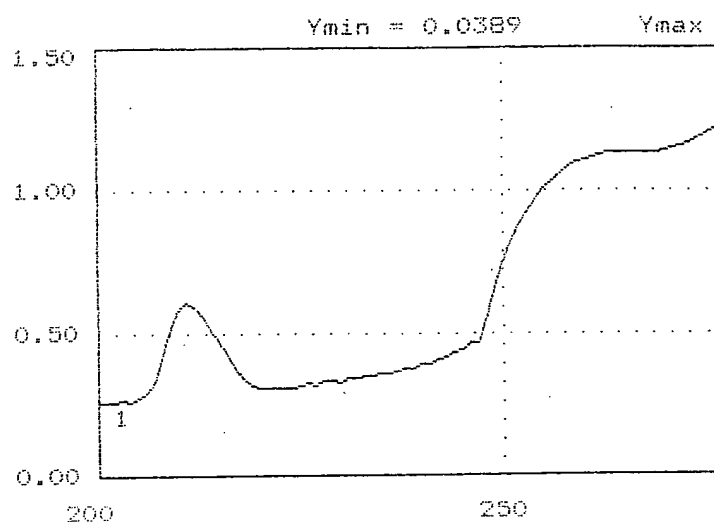
200 A / Tween 80

SOLUTION 150MG/L B CONTRE EAU

// 20-02-1996 11:25

SOLUTION 150MG/L B CONTRE TWEEN 80

// 20-02-1996 11:19



150 B / Tween

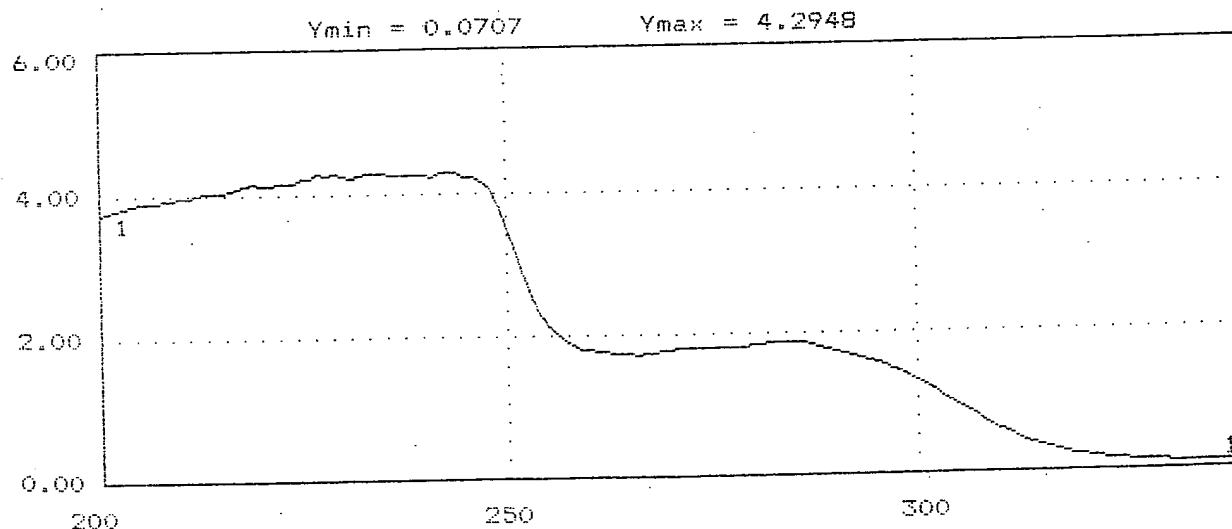
300.0	41	1.0579_1
299.0	42	1.1100_1
298.0	43	1.1563_1
297.0	44	1.1974_1
296.0	45	1.2327_1
295.0	46	1.2612_1
294.0	47	1.2825_1
293.0	48	1.3002_1
292.0	49	1.3131_1
291.0	50	1.3255_1
290.0	51	1.3311_1
289.0	52	1.3372_1
288.0	53	1.3381_1
287.0	54	1.3334_1
286.0	55	1.3242_1
285.0	56	1.3136_1
284.0	57	1.2981_1
283.0	58	1.2842_1
282.0	59	1.2713_1
281.0	60	1.2616_1
280.0	61	1.2555_1

CONTRON INSTRUMENTS

**FOURNIER 1001477**  
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SOLUTION 200MG/L A CONTRE EAU

// 20-02-1996 11:31



CONTRON INSTRUMENTS

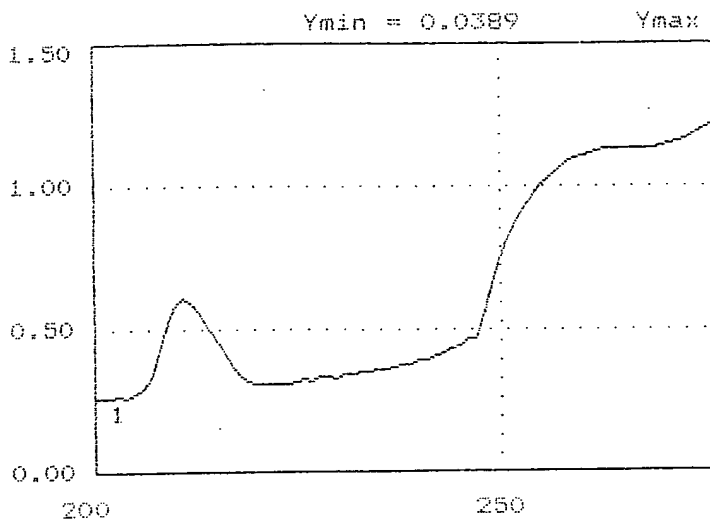
UVIKON 93C

SOLUTION 150MG/L B CONTRE EAU

20-02-1996 11:25

SOLUTION 150MG/L B CONTRE TWEEN 80

20-02-1996 11:19



300.0	41	1.0579_1
299.0	42	1.1100_1
298.0	43	1.1563_1
297.0	44	1.1974_1
296.0	45	1.2327_1
295.0	46	1.2612_1
294.0	47	1.2825_1
293.0	48	1.3002_1
292.0	49	1.3131_1
291.0	50	1.3255_1
290.0	51	1.3311_1
289.0	52	1.3372_1
288.0	53	1.3381_1
287.0	54	1.3334_1
286.0	55	1.3242_1
285.0	56	1.3136_1
284.0	57	1.2981_1
283.0	58	1.2842_1
282.0	59	1.2713_1
281.0	60	1.2616_1
280.0	61	1.2555_1

150 B / Tween

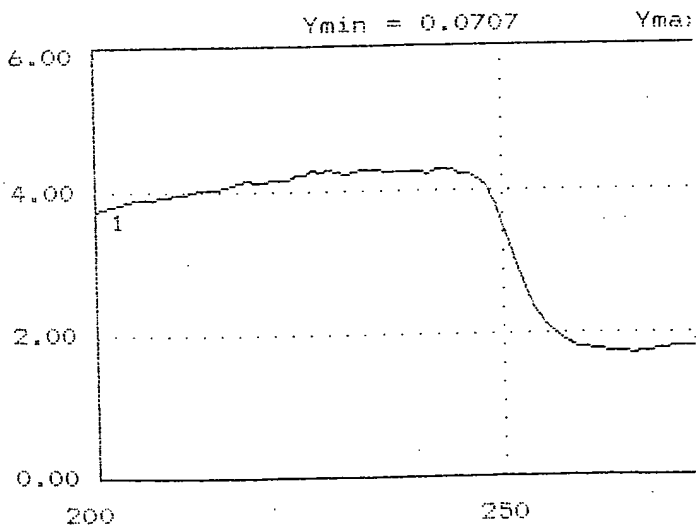
CONTRON INSTRUMENTS

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SOLUTION 200MG/L A CONTRE EAU

20-02-1996 11:31



300.0	41	1.2876_1
299.0	42	1.3514_1
298.0	43	1.4106_1
297.0	44	1.4630_1
296.0	45	1.5108_1
295.0	46	1.5580_1
294.0	47	1.5951_1
293.0	48	1.6302_1
292.0	49	1.6638_1
291.0	50	1.7000_1
290.0	51	1.7310_1
289.0	52	1.7649_1
288.0	53	1.7967_1
287.0	54	1.8216_1
286.0	55	1.8402_1
285.0	56	1.8527_1
284.0	57	1.8544_1
283.0	58	1.8496_1
282.0	59	1.8363_1
281.0	60	1.8233_1
280.0	61	1.8162_1

200 A / Eau

CONTRON INSTRUMENTS

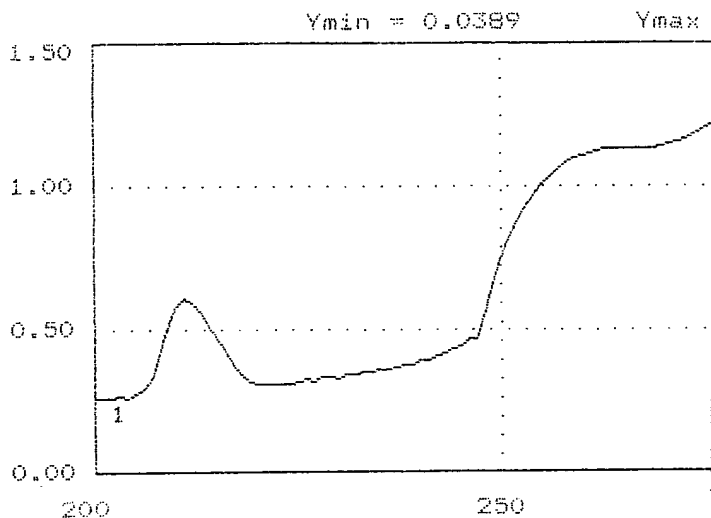
013

SOLUTION 150MG/L B CONTRE EAU

20-02-1996 11:25

SOLUTION 150MG/L B CONTRE TWEEN 80

20-02-1996 11:19



150 B / Tween

300.0	41	1.0579_1
299.0	42	1.1100_1
298.0	43	1.1583_1
297.0	44	1.1974_1
296.0	45	1.2327_1
295.0	46	1.2612_1
294.0	47	1.2825_1
293.0	48	1.3002_1
292.0	49	1.3131_1
291.0	50	1.3255_1
290.0	51	1.3311_1
289.0	52	1.3372_1
288.0	53	1.3381_1
287.0	54	1.3334_1
286.0	55	1.3242_1
285.0	56	1.3136_1
284.0	57	1.2981_1
283.0	58	1.2842_1
282.0	59	1.2713_1
281.0	60	1.2616_1
280.0	61	1.2555_1

ONTON INSTRUMENTS

FOURNIER 1001479

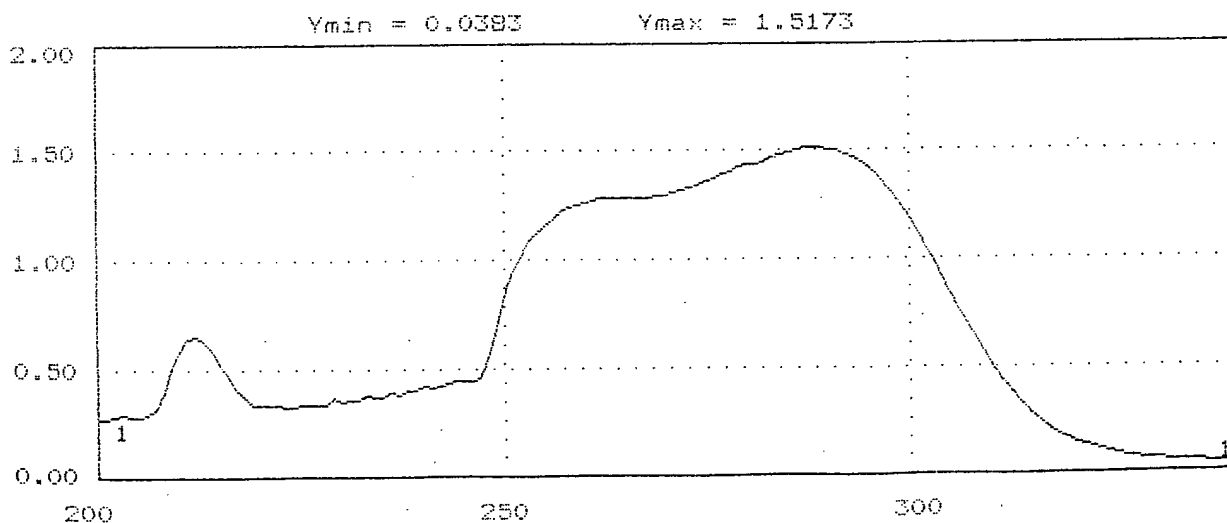
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Subject to  
Protective Order

SOLUTION 200MG/L A CONTRE EAU

20-02-1996 11:31

SOLUTION 200MG/L A CONTRE TWEEN 80

20-02-1996 13:31



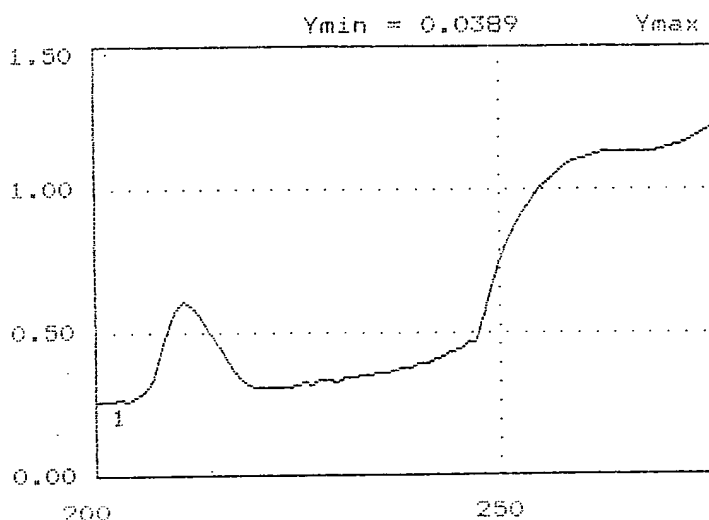
ONTON INSTRUMENTS

UVIKON 930

SOLUTION 150MG/L B CONTRE EAU

20-02-1996 11:25

SOLUTION 150MG/L B CONTRE TWEEN 80



20-02-1996 11:19

300.0	41	1.0579_1
299.0	42	1.1100_1
298.0	43	1.1563_1
297.0	44	1.1974_1
296.0	45	1.2327_1
295.0	46	1.2612_1
294.0	47	1.2825_1
293.0	48	1.3002_1
292.0	49	1.3131_1
291.0	50	1.3255_1
290.0	51	1.3311_1
289.0	52	1.3372_1
288.0	53	1.3381_1
287.0	54	1.3334_1
286.0	55	1.3242_1
285.0	56	1.3136_1
284.0	57	1.2981_1
283.0	58	1.2842_1
282.0	59	1.2713_1
281.0	60	1.2616_1
280.0	61	1.2555_1

150 B / Tween

MONTON INSTRUMENTS

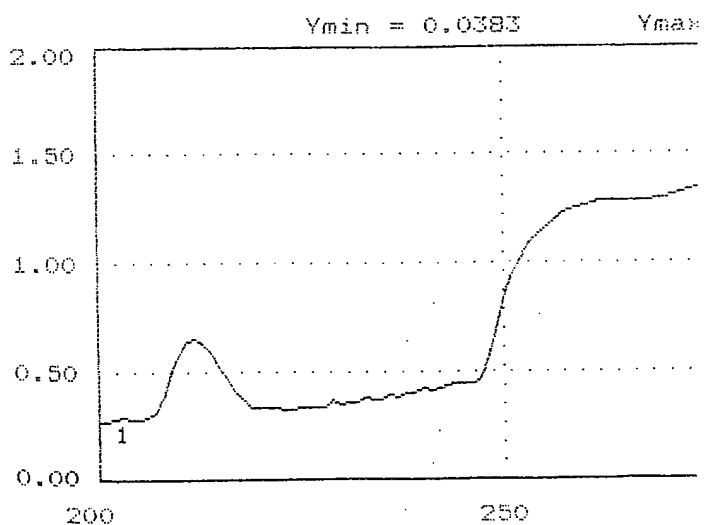
FOURNIER 1001480

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SOLUTION 200MG/L A CONTRE EAU

20-02-1996 11:31

SOLUTION 200MG/L A CONTRE TWEEN 80



20-02-1996 13:31

300.0	41	1.1792_1
299.0	42	1.2388_1
298.0	43	1.2925_1
297.0	44	1.3409_1
296.0	45	1.3827_1
295.0	46	1.4182_1
294.0	47	1.4447_1
293.0	48	1.4660_1
292.0	49	1.4816_1
291.0	50	1.4961_1
290.0	51	1.5044_1
289.0	52	1.5120_1
288.0	53	1.5144_1
287.0	54	1.5115_1
286.0	55	1.5031_1
285.0	56	1.4916_1
284.0	57	1.4764_1
283.0	58	1.4615_1
282.0	59	1.4465_1
281.0	60	1.4347_1
280.0	61	1.4318_1

200 A / Tween

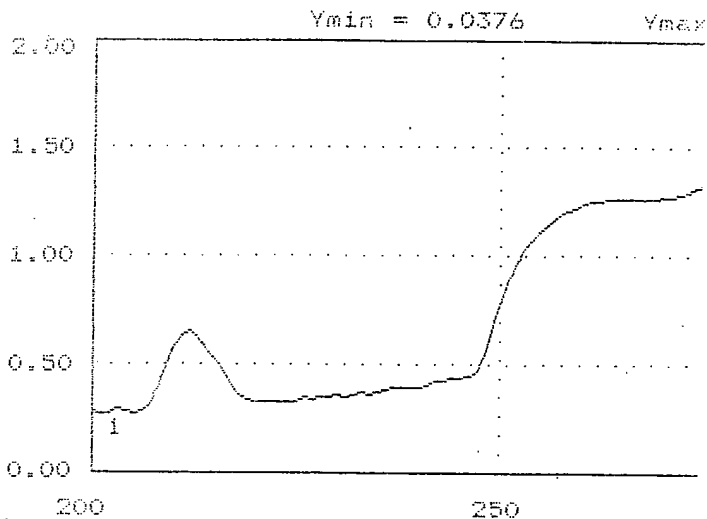
MONTON INSTRUMENTS

SOLUTION 200MB/L B CONTRE EAU

SOLUTION 200MB/L B CONTRE TWEEN 80

20-02-1996 13:4

20-02-1996 13:3



300.0 41 1.1781\_1  
299.0 42 1.2366\_1  
298.0 43 1.2895\_1  
297.0 44 1.3372\_1  
296.0 45 1.3768\_1  
295.0 46 1.4095\_1  
294.0 47 1.4338\_1  
293.0 48 1.4547\_1  
292.0 49 1.4695\_1  
291.0 50 1.4829\_1  
290.0 51 1.4905\_1  
289.0 52 1.4954\_1  
288.0 53 1.4976\_1  
287.0 54 1.4938\_1  
286.0 55 1.4837\_1  
285.0 56 1.4717\_1  
284.0 57 1.4539\_1  
283.0 58 1.4376\_1  
282.0 59 1.4242\_1  
281.0 60 1.4123\_1  
280.0 61 1.4082\_1

CONTRON INSTRUMENTS

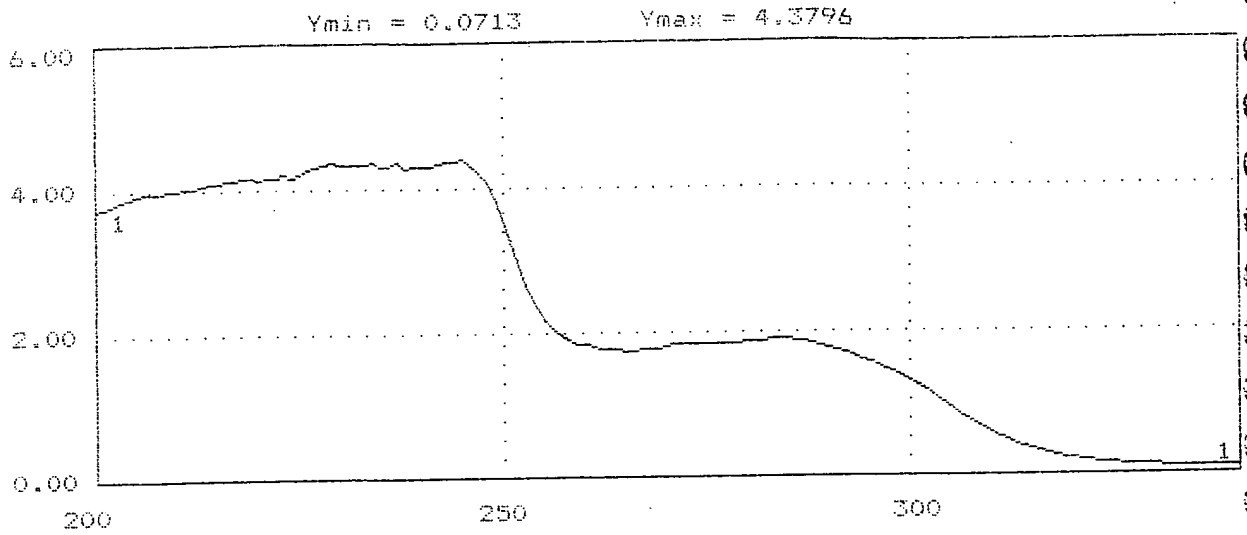
**FOURNIER 1001481**  
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Protective Order

6/3/97

014

SOLUTION 200MB/L B CONTRE EAU

//  
//20-02-1996 13:43



CONTRON INSTRUMENTS

UVIKON 930

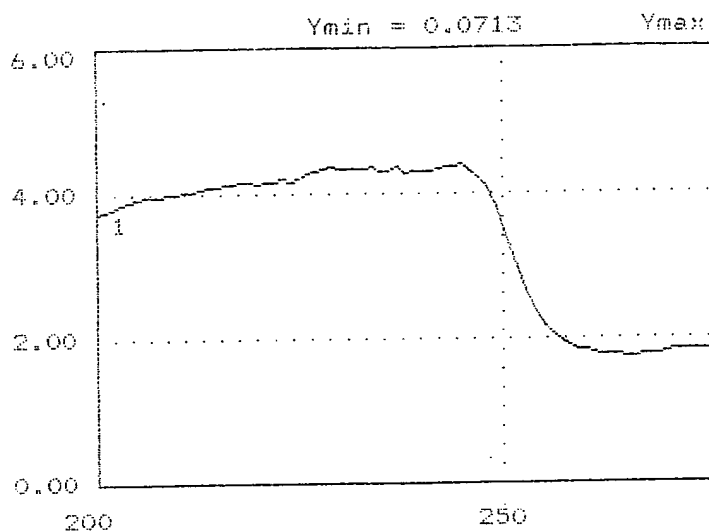
FOURNIER 1001482

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Protective Order

6/3/97

C14

SOLUTION 200MG/L B CONTRE EAU



CONTRON INSTRUMENTS

20-02-1996 13:43

200 B/scan

300.0	41	1.2851_1
299.0	42	1.3511_1
298.0	43	1.4130_1
297.0	44	1.4689_1
296.0	45	1.5187_1
295.0	46	1.5636_1
294.0	47	1.6036_1
293.0	48	1.6418_1
292.0	49	1.6772_1
291.0	50	1.7139_1
290.0	51	1.7475_1
289.0	52	1.7820_1
288.0	53	1.8145_1
287.0	54	1.8402_1
286.0	55	1.8607_1
285.0	56	1.8736_1
284.0	57	1.8744_1
283.0	58	1.8712_1
282.0	59	1.8610_1
281.0	60	1.8470_1
280.0	61	1.8423_1

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6/3/97



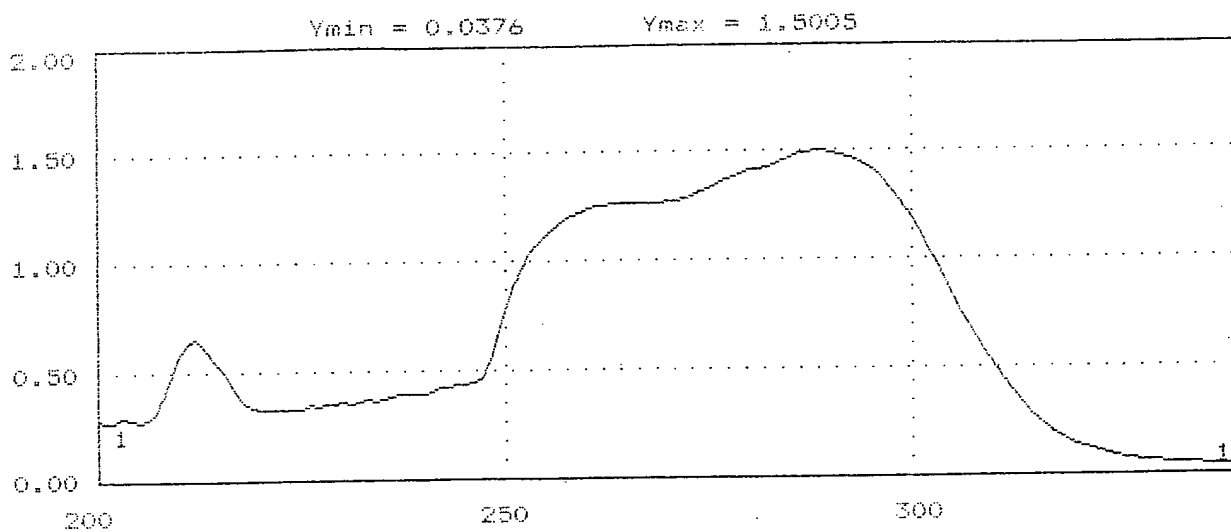
014

SOLUTION 200MG/L B CONTRE EAU

// 20-02-1996 13:43

SOLUTION 200MG/L B CONTRE TWEEN 80

// 20-02-1996 13:37



CONTRON INSTRUMENTS

UVIKON\_930

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•••

Ag 6/3/97

014

SOLUTION 200MG/L B CONTRE EAU

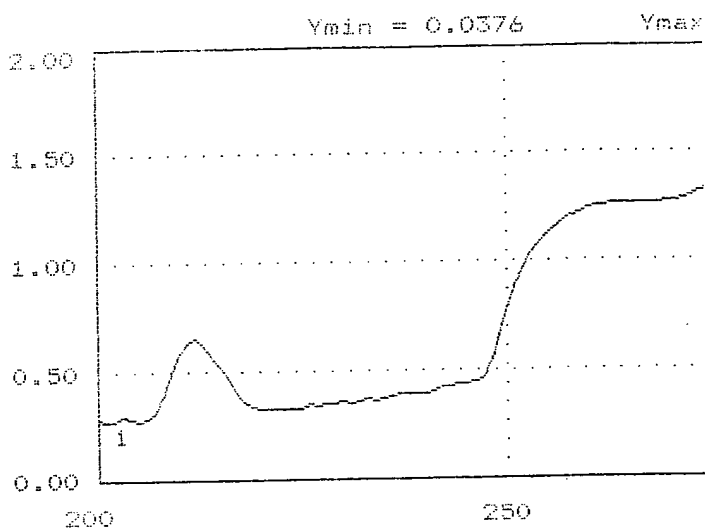
// 20-02-1996

13:43

SOLUTION 200MG/L B CONTRE TWEEN 80

// 20-02-1996

13:37



*100 B / Tween*

300.0	41	1.1781_1
299.0	42	1.2366_1
298.0	43	1.2895_1
297.0	44	1.3372_1
296.0	45	1.3768_1
295.0	46	1.4095_1
294.0	47	1.4338_1
293.0	48	1.4547_1
292.0	49	1.4695_1
291.0	50	1.4829_1
290.0	51	1.4905_1
289.0	52	1.4954_1
288.0	53	1.4976_1
287.0	54	1.4938_1
286.0	55	1.4837_1
285.0	56	1.4717_1
284.0	57	1.4539_1
283.0	58	1.4376_1
282.0	59	1.4242_1
281.0	60	1.4123_1
280.0	61	1.4082_1

CONTRON INSTRUMENTS

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6/3/97

**Droite étalon**  
Comiconrisat ARR 1709 dans tween 80 à 2%

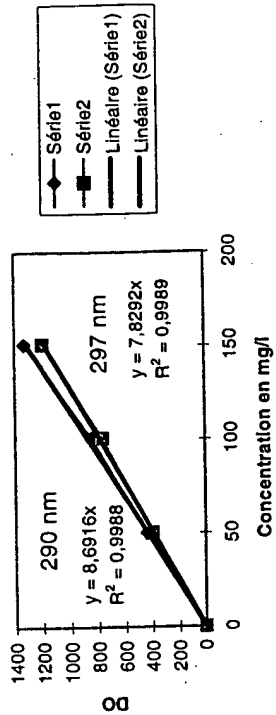
*Solutions B*

Conc mg/l	à 290nm	à 297 nm
0	0	0
50,24	443,2	401,4
100,97	849,1	766,2
151,21	1331,1	1197,4

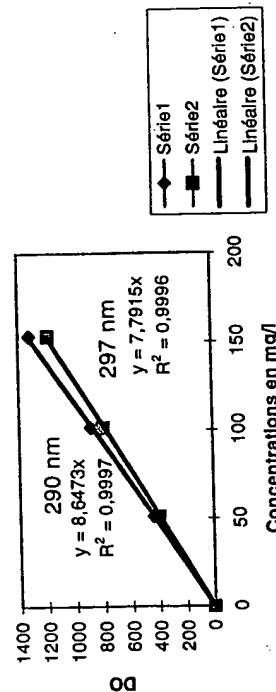
*Solutions A*

Conc mg/l	à 290nm	à 297 nm
0	0	0
50,72	441,4	401,9
101,45	889,8	803,2
153,62	1319,2	1186,3

**Droite étalon - Solutions B**



**Droite étalon - solutions A**



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m\cogetc\éno étalon

6/3/97

Validation des coefficients de DO :

$$\text{à } 230 \text{ nm} : \frac{8,6473}{8,6916} \times 100 = 99,5 \%$$

$$\text{le coefficient de DO est : } (8,6473 + 8,6916) / 2 = \boxed{8,67}$$

$$\text{à } 297 \text{ nm} : \frac{7,7915}{7,8292} \times 100 = 99,5 \%$$

$$\text{le coefficient de DO est : } (7,7915 + 7,8292) / 2 = \boxed{7,81}$$

Calcul de la concentration à saturation :

à partir des solutions à 1000 mg/l  
(analyse des résultats contre tween)

sol A			sol B		
230nm	297nm	$\lambda$	230nm	297nm	
1530,3	1360,1	DO	1530,3	1363,1	
176,5	174,1	conc mg/l	176,5	174,5	

à partir des solutions à 200 mg/l  
(analyse des résultats contre tween)

sol A			sol B		
230nm	297nm	$\lambda$	230nm	297nm	
1504,4	1340,9	DO	1490,5	1337,2	
173,5	171,7	conc mg/l	171,9	171,2	

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24/02/97

Gamme étalon

suite

017

Préparation de 2 solutions supplémentaires à 75 mg/l et 125 mg/l pour compléter et confirmer la gamme étalon déjà réalisée p15

### 1. Préparation du milieu

tween 80 à 2%.

tween Prolabo code 28 830 291 lot 065 FC.

eau purifiée du jour.

préparation pour les solutions de la gamme étalon et pour une dissolution.

eau: tare = 2951,6g      cc 24/02/97 M 24.02.97  
brut = 11163,8g      cc 24/02/97 M 24.02.97  
net = 8218,2g

tween:

d = 1,08 il faut que je rajoute 2% de tween =>

$$\begin{array}{l} 2 \longrightarrow 100 \\ x \longrightarrow 8218,2 + x \end{array}$$

$$2(8218,2 + x) = 100x$$

$$2 \times 8218,2 + 2x = 100x$$

$$16436,4 = 98x$$

$$x = 167,72$$

=> il faut rajouter 167,72 ml de tween 80  
soit 181,14g

tare = 11163,8g      cc 24/02/97 M 24.02.97

brut = remise à zéro      cc 24/02/97 M 24.02.97

net = 181,1g      cc 24/02/97 M 24.02.97

utilisation de la balance PC16 GAC III

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## 2. Pesée de la matière première

Cominoniser Féno / lauryl Sulfate de Na

ARR 1709

pour des concentrations en

Féno de :

75 mg/l

125 mg/l

pesée à réaliser en

Cominoniser :

77,63 mg/l soit 15,53 mg/200ml

129,38 mg/l soit 25,88 mg/200ml

24.02.97 15:37:21  
Code ARR 170924.02.97 15:43:46  
Code ARR 1709

ID 1

0.0000 g

85.4434 g T

0.0000 g

0.0163 g N

0.0000 g

-85.4597 g B

ID 2

0.0000 g

76.1364 g T

0.0000 g

0.0158 g N

0.0000 g

-76.1523 g B

ID 1

0.0000 g

85.5566 g T

0.0000 g

0.0256 g N

0.0000 g

-85.5823 g B

ID 2

0.0000 g

77.0217 g T

0.0000 g

0.0255 g N

0.0000 g

-77.0472 g B

Solution A :

$$16,3 \times 5 \times 200 / 207 = 78,74$$

$$16,3 \times 5 \times 200 / 207 = 84,35 \text{ mg/l}$$

$$25,6 \times 5 \times 200 / 207 = 123,67 \text{ mg/l}$$

Solution B :

$$15,8 \times 5 \times 200 / 207 = 76,33 \text{ mg/l}$$

$$25,5 \times 5 \times 200 / 207 = 123,19 \text{ mg/l}$$

les solutions sont placées sous agitation 1 nuit.

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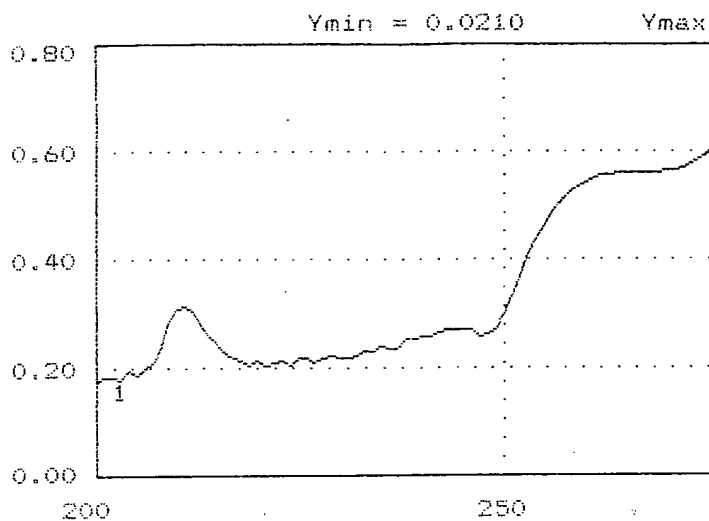
### 3. Réalisation de spectres

011

SOLUTION A 75 MG/L FEND - LBTWEEN

25-02-1996 13:5

SOLUTION A 75 MG/L FEND - LBTWEEN



25-02-1996 14:0

300.0	41	0.5337_1
299.0	42	0.5613_1
298.0	43	0.5835_1
297.0	44	0.6058_1
296.0	45	0.6240_1
295.0	46	0.6390_1
294.0	47	0.6492_1
293.0	48	0.6590_1
292.0	49	0.6682_1
291.0	50	0.6711_1
290.0	51	0.6729_1
289.0	52	0.6750_1
288.0	53	0.6757_1
287.0	54	0.6725_1
286.0	55	0.6670_1
285.0	56	0.6613_1
284.0	57	0.6525_1
283.0	58	0.6454_1
282.0	59	0.6374_1
281.0	60	0.6327_1
280.0	61	0.6289_1

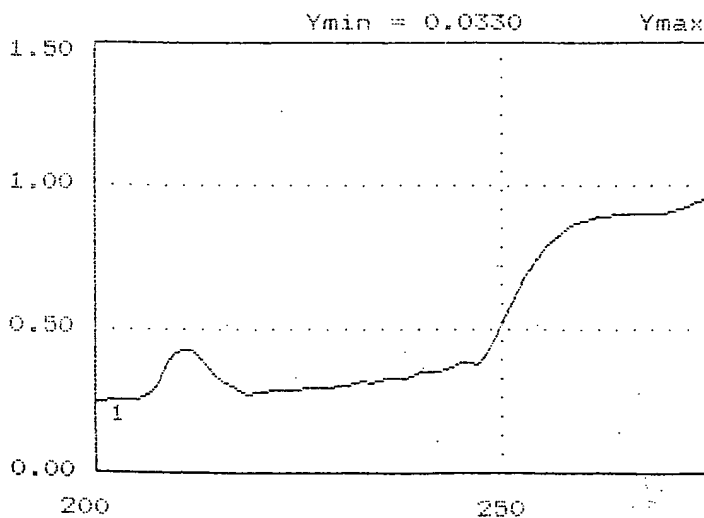
ONTRON INSTRUMENTS

FOURNIER 1001490

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SOLUTION A 125 MG/L FEND - LBTWEEN

SOLUTION A 125 MG/L FEND - LBTWEEN



25-02-1996 14:15

300.0	41	0.8433_1
299.0	42	0.8867_1
298.0	43	0.9226_1
297.0	44	0.9577_1
296.0	45	0.9862_1
295.0	46	1.0103_1
294.0	47	1.0275_1
293.0	48	1.0429_1
292.0	49	1.0533_1
291.0	50	1.0630_1
290.0	51	1.0669_1
289.0	52	1.0709_1
288.0	53	1.0716_1
287.0	54	1.0686_1
286.0	55	1.0605_1
285.0	56	1.0505_1
284.0	57	1.0375_1
283.0	58	1.0264_1
282.0	59	1.0149_1
281.0	60	1.0076_1
280.0	61	1.0020_1

ONTRON INSTRUMENTS

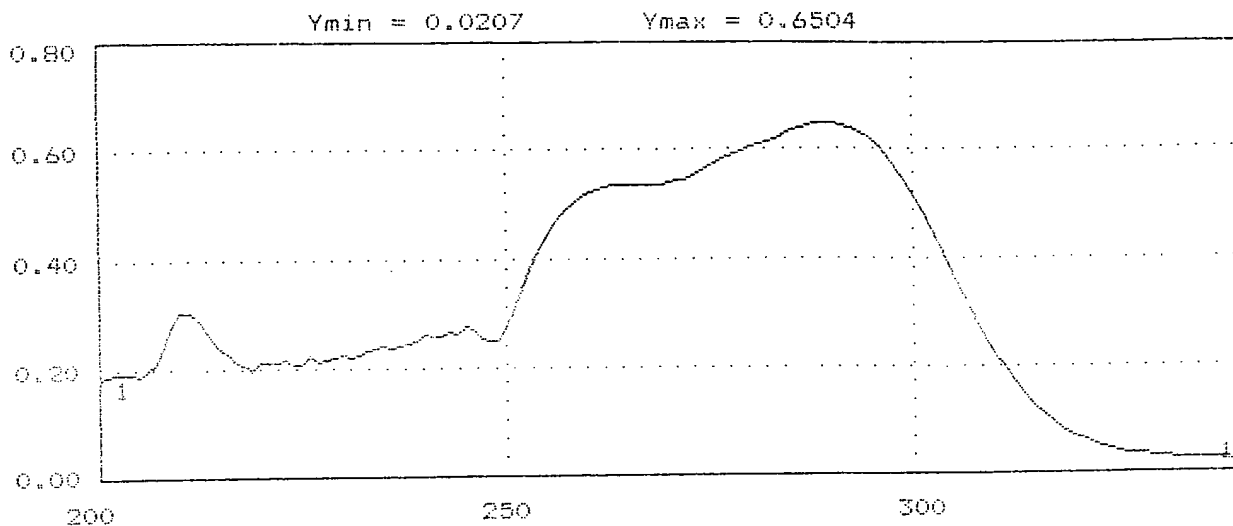
### 3. Réalisation de spectres.

019

SOLUTION A 75 MG/L FENO - LBETWEEN

AD

25-02-1996 13:55

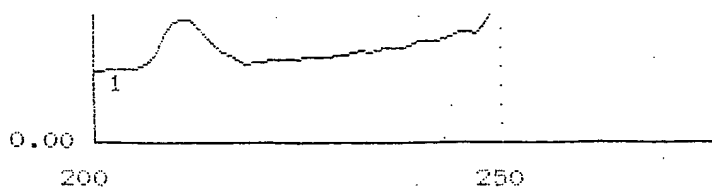


KONTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001491

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KONTRON INSTRUMENTS

288.0	53	1.0716_1
287.0	54	1.0686_1
286.0	55	1.0605_1
285.0	56	1.0505_1
284.0	57	1.0375_1
283.0	58	1.0264_1
282.0	59	1.0149_1
281.0	60	1.0076_1
280.0	61	1.0020_1

UF

0

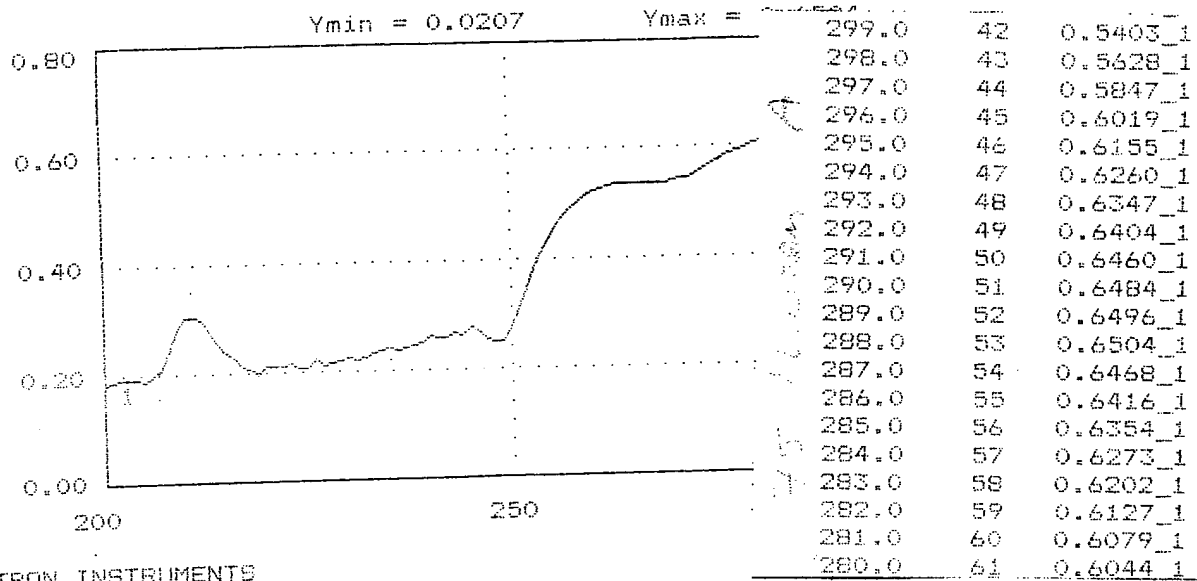


### 3. Réalisation de spectres.

019

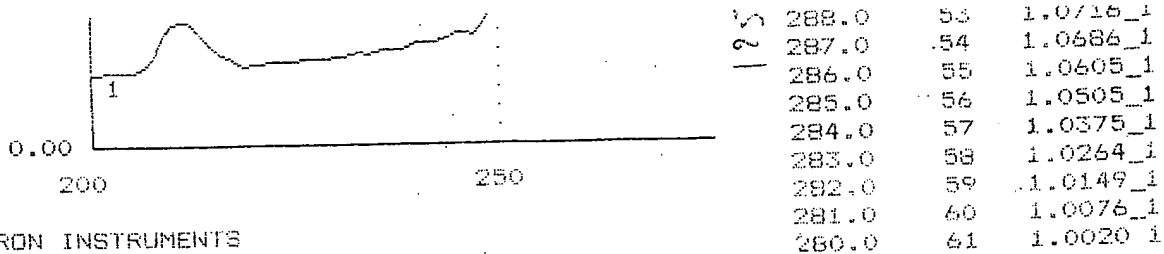
SOLUTION A 75 MG/L FENO - LBWEEN

25-02-1996 13:55



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.. Réalisation de spectres ..

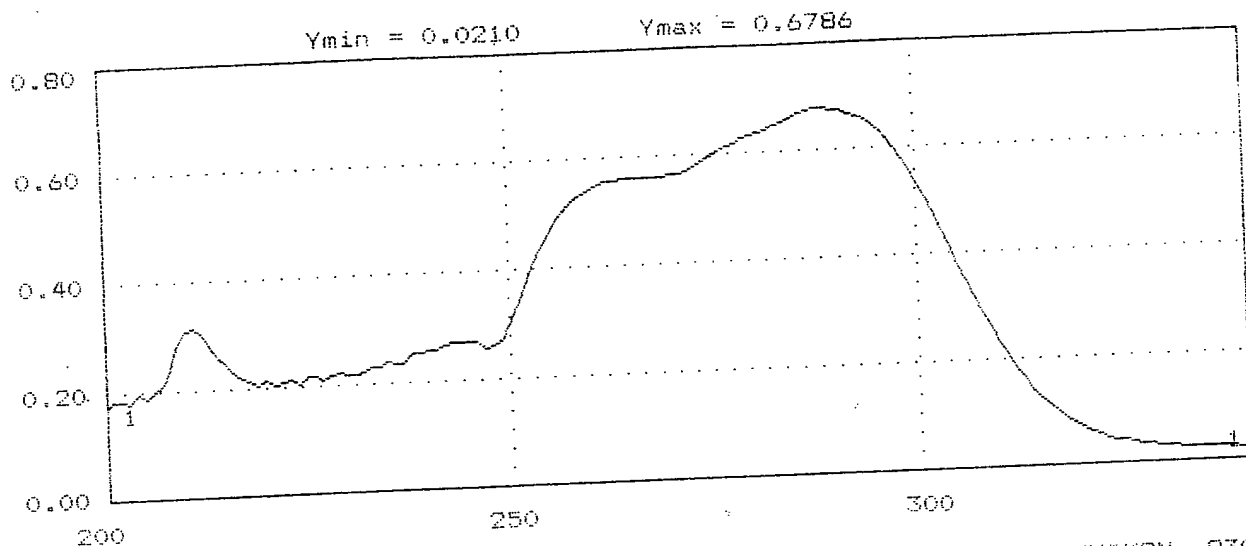
C 10

SOLUTION A 75 MG/L FEND - LBTWEEN

25-02-1996 13:55

SOLUTION A 75 MG/L FEND - LBTWEEN

25-02-1996 14:01



ITRON INSTRUMENTS

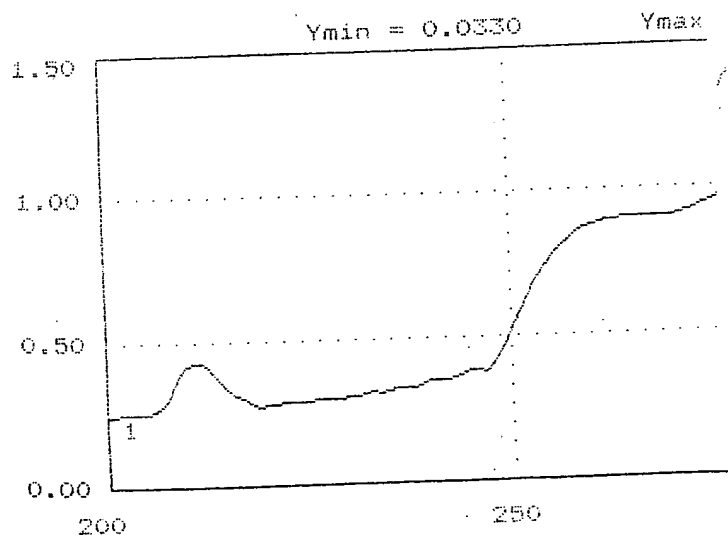
SOLUTION A 125 MG/L FEND - LBTWEEN

(A)

FOURNIER 1001493

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Subject to  
Protective Order

SOLUTION A 125 MG/L FEND - LBTWEEN



125 MG/L

293.0	48	1.0429_1
292.0	49	1.0533_1
291.0	50	1.0630_1
290.0	51	1.0669_1
289.0	52	1.0709_1
288.0	53	1.0716_1
287.0	54	1.0686_1
286.0	55	1.0605_1
285.0	56	1.0505_1
284.0	57	1.0375_1
283.0	58	1.0264_1
282.0	59	1.0149_1
281.0	60	1.0076_1
280.0	61	1.0020_1

ITRON INSTRUMENTS

### 3. Réalisation de spectres

004  
010

SOLUTION A 75 MG/L FEND - LBTWEEN

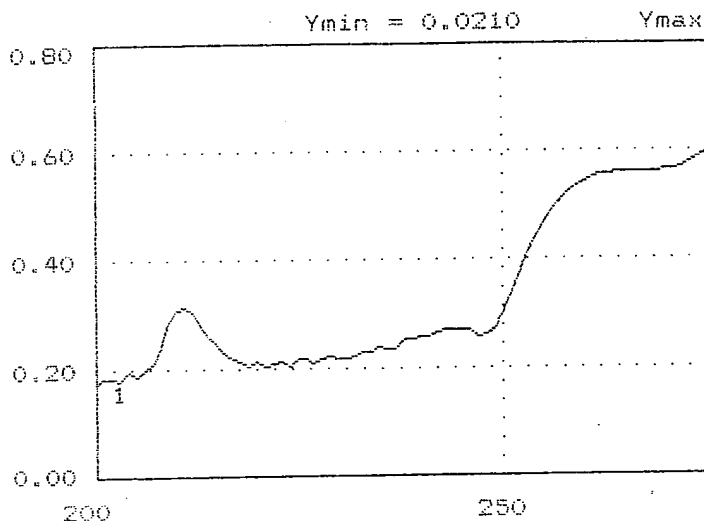
(A)

25-02-1996 13:55

SOLUTION A 75 MG/L FEND - LBTWEEN

(B)

25-02-1996 14:01



300.0	41	0.5337_1
299.0	42	0.5613_1
298.0	43	0.5835_1
297.0	44	0.6058_1
296.0	45	0.6240_1
295.0	46	0.6390_1
294.0	47	0.6492_1
293.0	48	0.6590_1
292.0	49	0.6652_1
291.0	50	0.6711_1
290.0	51	0.6729_1
289.0	52	0.6750_1
288.0	53	0.6757_1
287.0	54	0.6725_1
286.0	55	0.6670_1
285.0	56	0.6613_1
284.0	57	0.6525_1
283.0	58	0.6454_1
282.0	59	0.6374_1
281.0	60	0.6327_1
280.0	61	0.6289_1

ONTRON INSTRUMENTS

FOURNIER 1001494

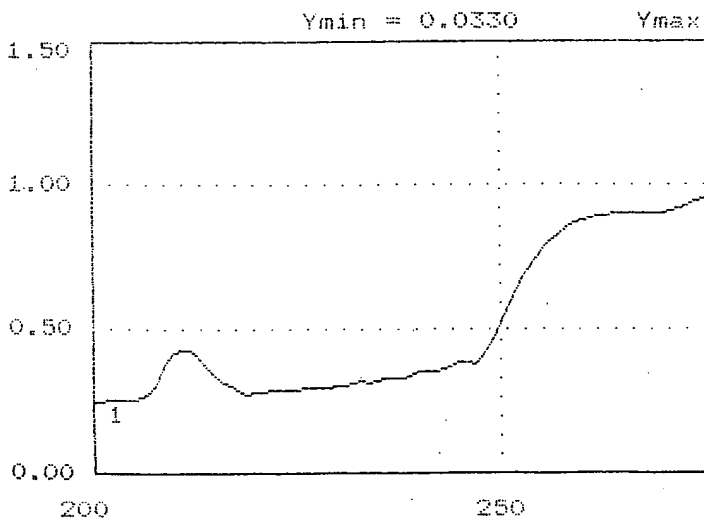
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Subject to  
Protective Order

SOLUTION A 125 MG/L FEND - LBTWEEN

(A)

SOLUTION A 125 MG/L FEND - LBTWEEN

25-02-1996 14:19



300.0	41	0.8433_1
299.0	42	0.8867_1
298.0	43	0.9226_1
297.0	44	0.9577_1
296.0	45	0.9862_1
295.0	46	1.0103_1
294.0	47	1.0275_1
293.0	48	1.0429_1
292.0	49	1.0533_1
291.0	50	1.0630_1
290.0	51	1.0669_1
289.0	52	1.0709_1
288.0	53	1.0716_1
287.0	54	1.0686_1
286.0	55	1.0605_1
285.0	56	1.0505_1
284.0	57	1.0375_1
283.0	58	1.0264_1
282.0	59	1.0149_1
281.0	60	1.0076_1
280.0	61	1.0000_1

ONTRON INSTRUMENTS

### 3. Réalisation de spectres

021

019

SOLUTION A 75 MG/L FEND - LBTWEEN

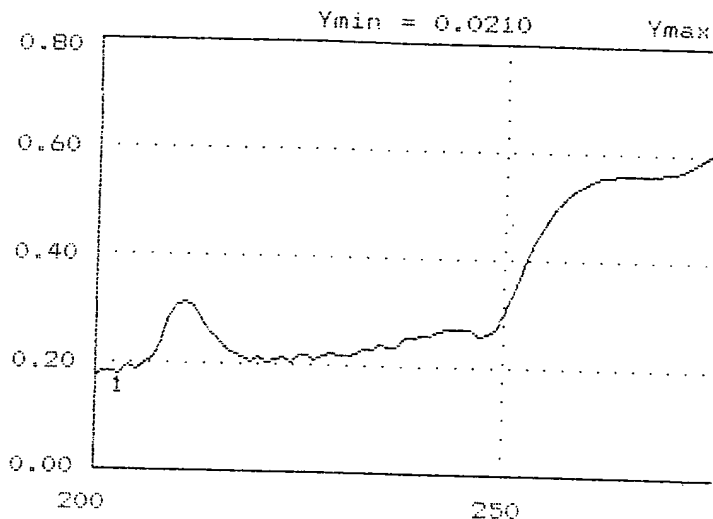
(A)

25-02-1996 13:55

SOLUTION A 75 MG/L FEND - LBTWEEN

(B)

25-02-1996 14:01



300.0	41	0.5337_1
299.0	42	0.5613_1
298.0	43	0.5835_1
297.0	44	0.6058_1
296.0	45	0.6240_1
295.0	46	0.6390_1
294.0	47	0.6492_1
293.0	48	0.6590_1
292.0	49	0.6652_1
291.0	50	0.6711_1
290.0	51	0.6729_1
289.0	52	0.6750_1
288.0	53	0.6757_1
287.0	54	0.6725_1
286.0	55	0.6670_1
285.0	56	0.6613_1
284.0	57	0.6525_1
283.0	58	0.6454_1
282.0	59	0.6374_1
281.0	60	0.6327_1
280.0	61	0.6289_1

INTRON INSTRUMENTS

FOURNIER 1001495

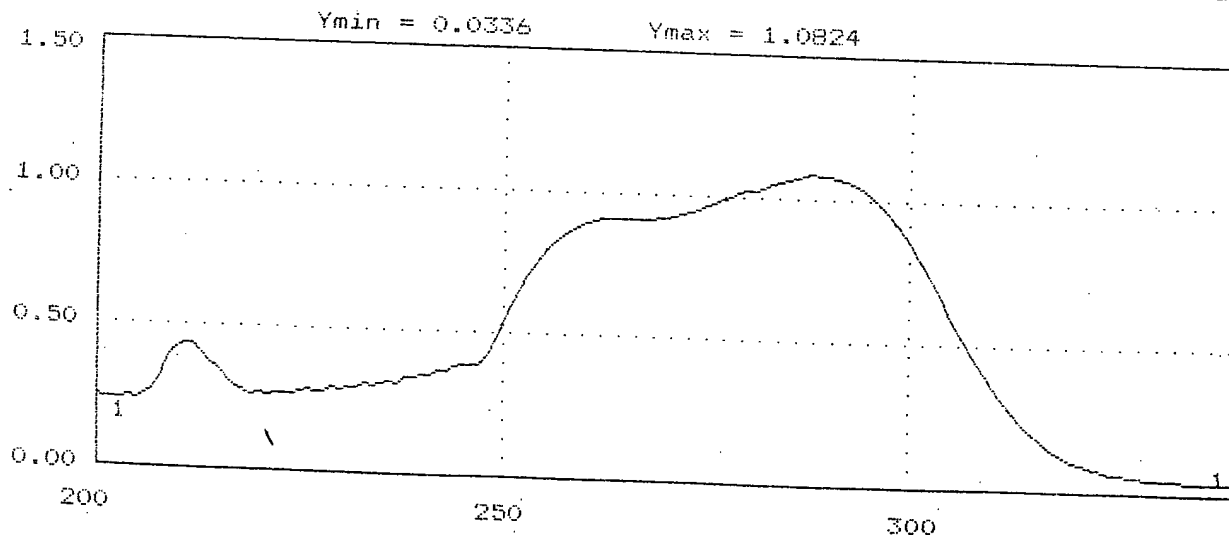
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Subject to  
Protective Order

3

SOLUTION A 125 MG/L FEND - LBTWEEN

(A)

25-02-1996 14:13



RON INSTRUMENTS

UVIKON 930

### 3. Realization de spectres

021

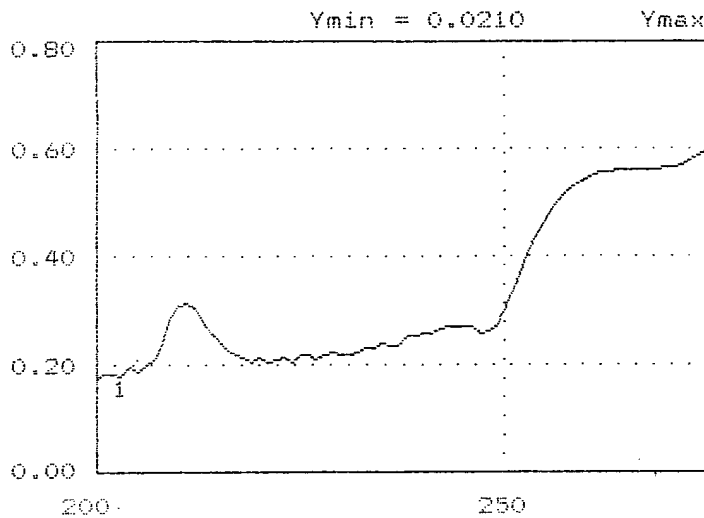
019

SOLUTION A 75 MG/L FEND - LBTWEEN (A)

25-02-1996 13:55

SOLUTION A 75 MG/L FEND - LBTWEEN (B)

25-02-1996 14:01



300.0	41	0.5337_1
299.0	42	0.5613_1
298.0	43	0.5835_1
297.0	44	0.6058_1
296.0	45	0.6240_1
295.0	46	0.6390_1
294.0	47	0.6492_1
293.0	48	0.6590_1
292.0	49	0.6652_1
291.0	50	0.6711_1
290.0	51	0.6729_1
289.0	52	0.6750_1
288.0	53	0.6757_1
287.0	54	0.6725_1
286.0	55	0.6670_1
285.0	56	0.6613_1
284.0	57	0.6525_1
283.0	58	0.6454_1
282.0	59	0.6374_1
281.0	60	0.6327_1
280.0	61	0.6289_1

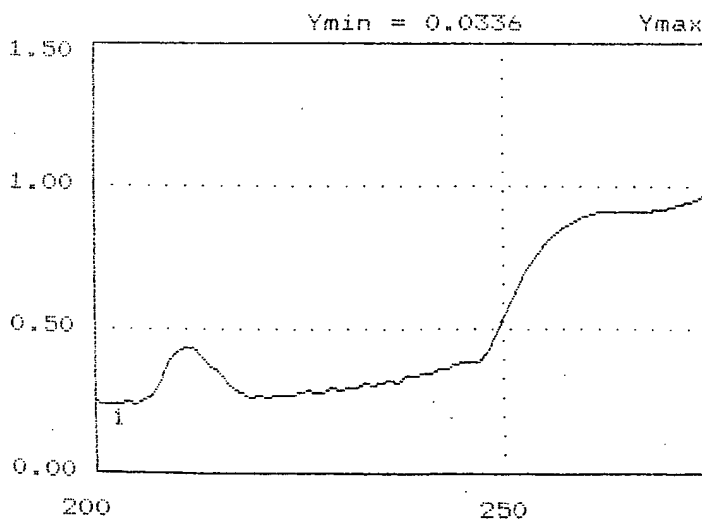
ONTRON INSTRUMENTS

FOURNIER 1001496

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SOLUTION A 125 MG/L FEND - LBTWEEN (A)

25-02-1996 14:13



300.0	41	0.8433_1
299.0	42	0.8860_1
298.0	43	0.9234_1
297.0	44	0.9590_1
296.0	45	0.9887_1
295.0	46	1.0131_1
294.0	47	1.0308_1
293.0	48	1.0469_1
292.0	49	1.0580_1
291.0	50	1.0681_1
290.0	51	1.0738_1
289.0	52	1.0778_1
288.0	53	1.0795_1
287.0	54	1.0770_1
286.0	55	1.0701_1
285.0	56	1.0604_1
284.0	57	1.0483_1
283.0	58	1.0380_1
282.0	59	1.0270_1
281.0	60	1.0191_1
280.0	61	1.0146_1

ONTRON INSTRUMENTS

130

### 3. Réalisation de spectres.

019

SOLUTION A 75 MG/L FEND - LBTWEEN

(A)

25-02-1996

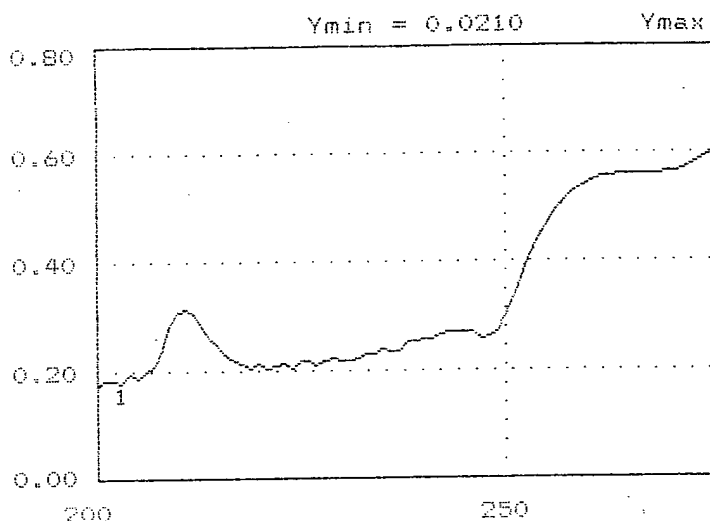
13:55

SOLUTION A 75 MG/L FEND - LBTWEEN

(B)

25-02-1996

14:01



300.0	41	0.5337_1
299.0	42	0.5613_1
298.0	43	0.5835_1
297.0	44	0.6058_1
296.0	45	0.6240_1
295.0	46	0.6390_1
294.0	47	0.6492_1
293.0	48	0.6590_1
292.0	49	0.6652_1
291.0	50	0.6711_1
290.0	51	0.6729_1
289.0	52	0.6750_1
288.0	53	0.6757_1
287.0	54	0.6725_1
286.0	55	0.6670_1
285.0	56	0.6613_1
284.0	57	0.6525_1
283.0	58	0.6454_1
282.0	59	0.6374_1
281.0	60	0.6327_1
280.0	61	0.6289_1

CONTRON INSTRUMENTS

FOURNIER 1001497

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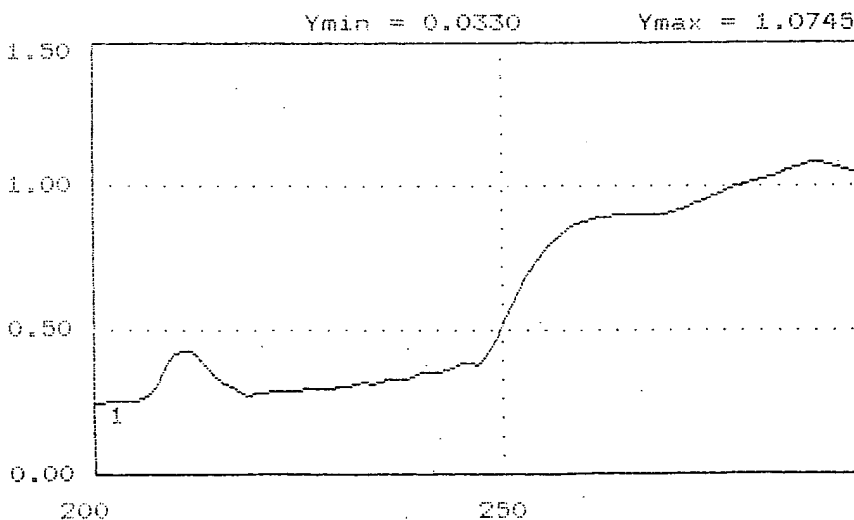
SOLUTION A 125 MG/L FEND - LBTWEEN

(A)

SOLUTION A 125 MG/L FEND - LBTWEEN

25-02-1996

14:19



CONTRON INSTRUMENTS

UVIKON 930

### 3. Réalisation des spectres

019

SOLUTION A 75 MG/L FENO - LBTWEEN

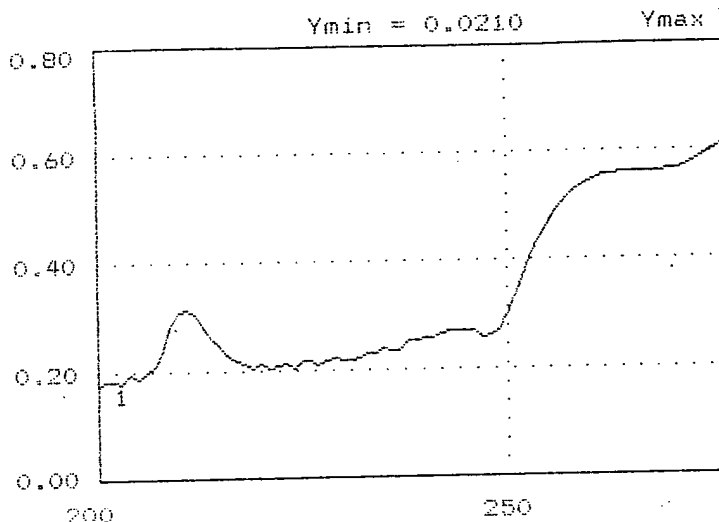
(A)

25-02-1996 13:55

SOLUTION A 75 MG/L FENO - LBTWEEN

(B)

25-02-1996 14:01



75 Tween (B)

300.0	41	0.5337_1
299.0	42	0.5613_1
298.0	43	0.5835_1
297.0	44	0.6058_1
296.0	45	0.6240_1
295.0	46	0.6390_1
294.0	47	0.6492_1
293.0	48	0.6590_1
292.0	49	0.6652_1
291.0	50	0.6711_1
290.0	51	0.6729_1
289.0	52	0.6750_1
288.0	53	0.6757_1
287.0	54	0.6725_1
286.0	55	0.6670_1
285.0	56	0.6613_1
284.0	57	0.6525_1
283.0	58	0.6454_1
282.0	59	0.6374_1
281.0	60	0.6327_1
280.0	61	0.6289_1

ONTRON INSTRUMENTS

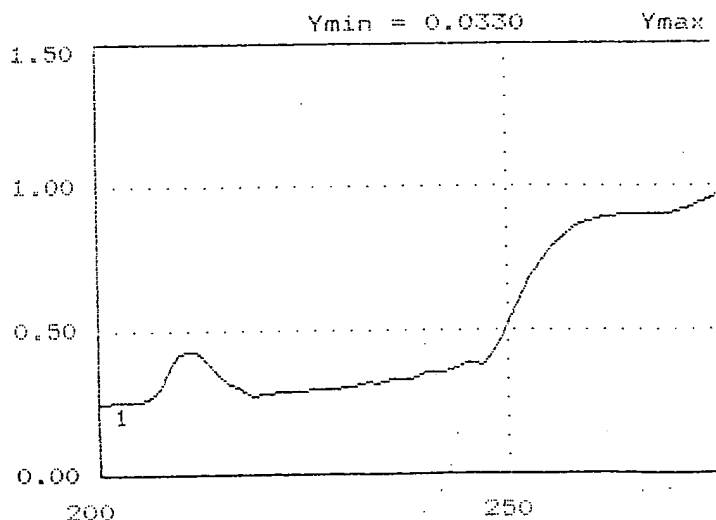
**FOURNIER 1001498**  
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Subject to  
Protective Order

SOLUTION A 125 MG/L FENO - LBTWEEN

(A)

SOLUTION A 125 MG/L FENO - LBTWEEN

25-02-1996 14:19



125 Tween (B)

300.0	41	0.8433_1
299.0	42	0.8867_1
298.0	43	0.9226_1
297.0	44	0.9577_1
296.0	45	0.9862_1
295.0	46	1.0103_1
294.0	47	1.0275_1
293.0	48	1.0429_1
292.0	49	1.0533_1
291.0	50	1.0630_1
290.0	51	1.0669_1
289.0	52	1.0709_1
288.0	53	1.0716_1
287.0	54	1.0686_1
286.0	55	1.0605_1
285.0	56	1.0505_1
284.0	57	1.0375_1
283.0	58	1.0264_1
282.0	59	1.0149_1
281.0	60	1.0076_1
280.0	61	1.0020_1

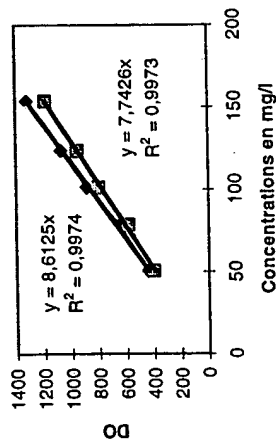
ONTRON INSTRUMENTS

**Droite étalon**  
Comiconisat ARR 1709 dans tween 80 à 2%

Solutions A

Conc mg/l	à 290nm	à 297 nm
50,72	441,4	401,9
78,74	648,4	584,7
101,45	889,8	803,2
123,67	1073,8	959
153,62	1319,2	1186,3

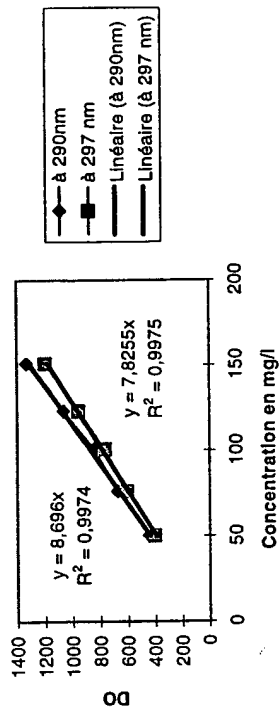
Droite étalon - Solutions A



Solutions B

Conc mg/l	à 290nm	à 297 nm
50,24	443,2	401,4
76,33	672,9	605,8
100,97	849,1	766,2
123,19	1066,9	957,7
151,21	1331,1	1197,4

Droite étalon - Solutions B



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# Vérification des coefficients de DO.

calcul à 290 nm :

$$100 \times \frac{8,6125}{8,696} = 99,0\%$$

on prendra comme coefficient de DO :

$$(8,6125 + 8,696) / 2 = \boxed{8,65}$$

Calcul de la concentration à saturation avec le coefficient de DO de 8,65.

solutions à 1000 mg/ml

(reprise de données de la page 016)

solutions à 200 mg/l

à 290 nm - (A)

$$DO = 1530,3$$

$$CS = 176,9$$

(B)

$$DO = 1530,3$$

$$CS = 176,9$$

(A)

$$DO = 1504,4$$

$$CS = 173,9$$

(B)

$$DO = 1490,5$$

$$CS = 172,3$$

## Conclusion :

la concentration à saturation du fénofibrate dans le tween 80 à 2% est 176,9 mg/l.

Dans le cahier lipantyl 200 comprimé n°2 page 38, la concentration à saturation du fénofibrate dans le LSNa 0,02M est 163,6 mg/l.

Les 2 cs sont comparables.

022

Dissolution1) Nature première :

Comprimés de Fénofibrate 100mg.

lot # : 340. date 02/07/97.

Pharma PASS.

2) Préparation du milieu de dissolution

voir p017.

3) Pesée des bols de dissolution

1 l de milieu

	Remise à géro	Pesée	Signature
1			
2			
3			
4			
5			
6			

FOURNIER 1001501

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Protective Order

26.02.97.

Préparation d'une solution  
avec éthanol.

023

+33 3 88 66 35 42 P.2

24.FEV'97 10:44 PHARMA PASS

Page n°: 1/1  
Analyse n°: 1086  
Lot n°: Capsules Fenofibrate N: 346  
Projet n°: 23  
23-3  
Termin  
Dissoudre  $\approx 28$  mg de fenofibrate dans  
50 ml d'éthanol absolu: colorer 5/50 ml  
avec eau + 1,25% Tween 80  $(\Rightarrow) \approx 56$  mg/l  
Filtres 0,7  $\mu$ m  
X: témoin: 400.0 - 200.0 nm: pts 201: int 1.00: ord -3.982 - 6.0000 A  
Inf: témoin fenofibrate 66.9 mg/l eau + 1.25% tween 80, cellule: 1.00cm  
PRAY MAY: tuteur a aaaa

FOURNIER 1001502

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Protective Order

Page n°: 1/4

Analyse n°: 1086

Projet n°: 23

- eau + 1,25% Tween 80 : 1200ml

- pilule, 50t/mk

- d: 0,2cm.

(n: 313)

Lot n°: Capsule Fenofibrate

N: 366

Témoin

disperse  $\approx$  28 mg de fenofibrate dans

50 ml d'éthanol absolu: diluer 5/50 ml

avec eau + 1,25% Tween 80

( $\approx$ ) 256 mg/l

filtres

0,7  $\mu$ m

X: témoin: 400.0 - 200.0 nm; pts 201; int 1.00; ord -3.982 - 6.0000 A  
Inf: témoin fenofibrate 66.9 mg/l eau+1.25%tween 80, cellule:1.00cm  
PEAK MAX: THRESH 0.0100

nm	A	nm	A	nm	A
201.0	6.0000	243.0	2.8408		
204.0	1.0036	245.0	3.9927		
206.0	1.9106	247.0	5.5934		
209.0	0.2426	252.0	3.7172		
211.0	6.0000	256.0	5.1515		
213.0	5.1762	→ 265.0	0.6828		
216.0	-0.0907	→ 279.0	0.7833		
219.0	5.7375	→ 297.0	1.9943		
222.0	-0.4548				
226.0	5.7249				
228.0	4.0887				
231.0	2.4671				
236.0	6.0000				
240.0	6.0000				

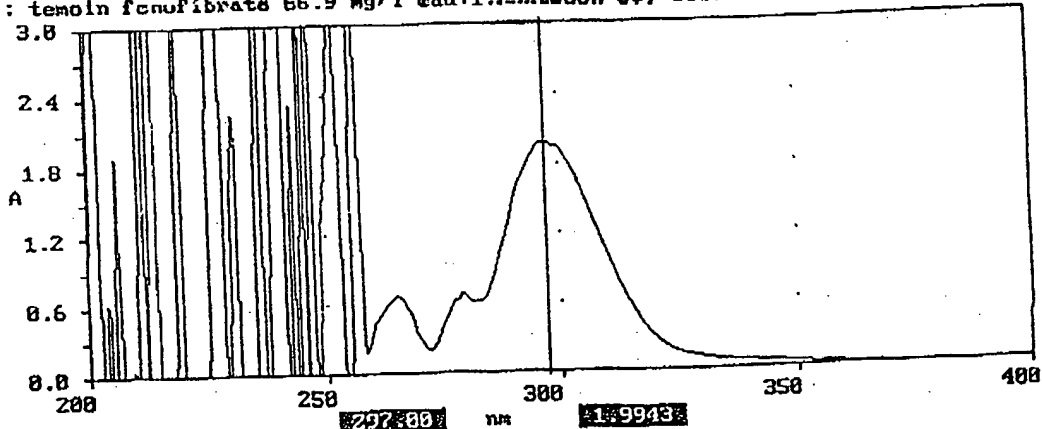
X: témoin: 400.0 - 200.0 nm; pts 201; int 1.00; ord -3.982 - 6.0000 A  
Inf: témoin fenofibrate 66.9 mg/l eau+1.25%tween 80, cellule:1.00cm  
PEAK MIN: THRESH 0.0100

nm	A	nm	A	nm	A
203.0	-2.2548	244.0	-1.8215		
205.0	-0.8260	246.0	-0.6235		
208.0	-3.9829	248.0	-2.1745		
210.0	-0.7655	254.0	-0.7590		
212.0	-1.7086	258.0	0.1563		
215.0	-1.7906	→ 272.0	0.2022		
217.0	-0.9112	→ 281.0	0.6201		
220.0	-1.7050				
224.0	-1.6215				
227.0	3.2646				
229.0	-2.1676				
234.0	-1.7042				
237.0	-0.7224				
242.0	-2.1051				

FOURNIER 1001503

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X: témoin: 400.0 - 200.0 nm; pts 201; int 1.00; ord -3.982 - 6.0000 A  
Inf: témoin fenofibrate 66.9 mg/l eau+1.25%tween 80, cellule:1.00cm



6/19/95

Date:

x préparation de tween 1,25 %.

prélèvement à la pipette graduée de 12,5 ml de tween 20  
référence N° lot 215 GC. dans une fiole de 1 l - qs à  
l'eau purifiée -

x préparation d'une solution de tween 1,25 % avec 1/10 éthanol  
dans une fiole de 100 ml, introduction à la pipette à  
2 traits de 10 ml d'éthanol - qs avec la solution de  
tween à 1,25 %.

x préparation de la solution de pénicilline.

pesée de 28,38 mg de Comprimés ARR 1709 dans une  
fiole de 50 ml - solubilisation et qs avec l'éthanol

26.02.97

09:07:50

Code

1709

0.0000 g

37.3198 g

0.0000 g

0.0289 g

0.0000 g

-37.3489 g

// pesée sur balance AB204 GALL205.

Transvasement de 50 ml de solution obtenus dans une  
fiole de 500 ml - qs avec la solution de tween 20 à 1,25 %.  
Homogénéisation.

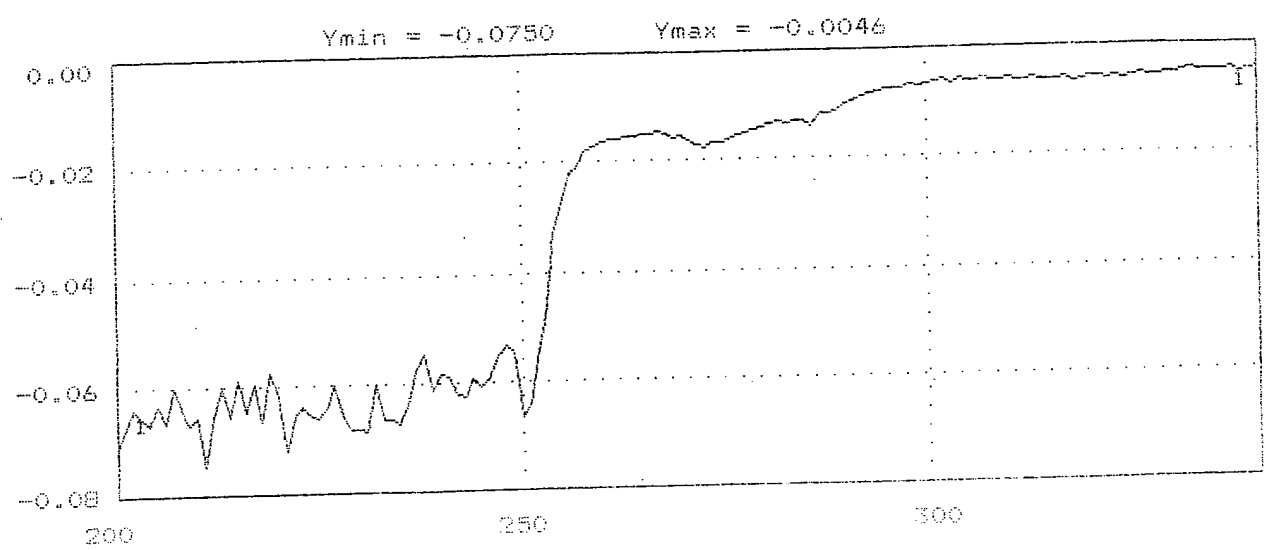
FOURNIER 1001504

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Protective Order

025

LIGNE DE BASE TWEEN 1.25 POUR 100 + ETHANOL 1/10 (CUVE DE 1CM)

26-02-1996 10:28



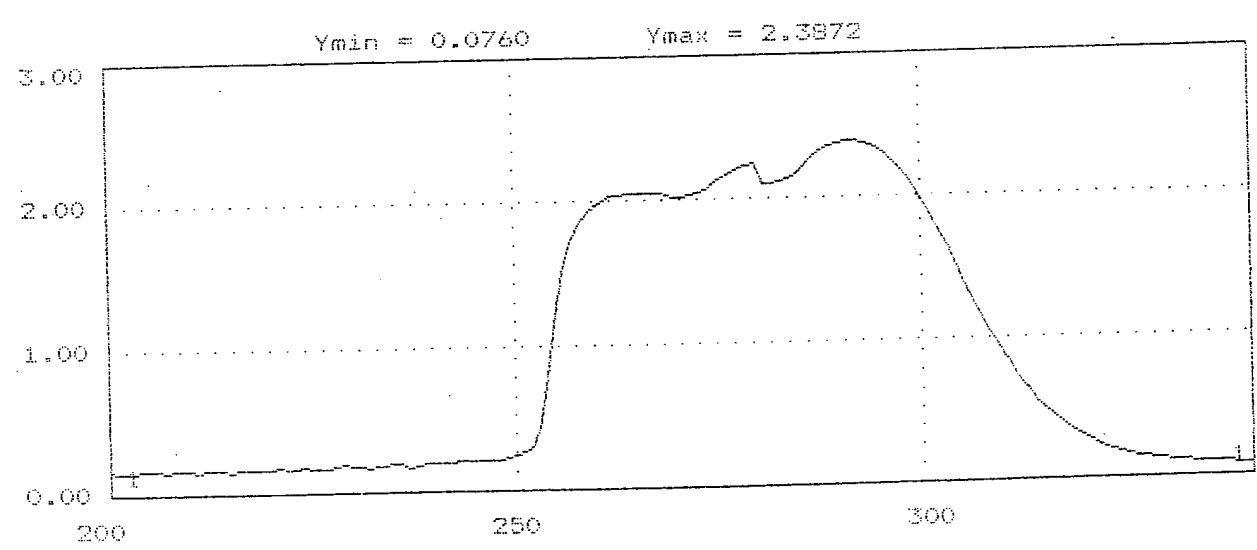
KONTRON INSTRUMENTS

UVIKON 930

LIGNE DE BASE TWEEN 1.25 POUR 100 + ETHANOL 1/10 (CUVE DE 1CM)

FENO à 56mg/l dans

26-02-1996 10:34



KONTRON INSTRUMENTS

UVIKON 930

obtention d'un spectre qui ne ressemble toujours pas à celui obtenu par Pharma Pass

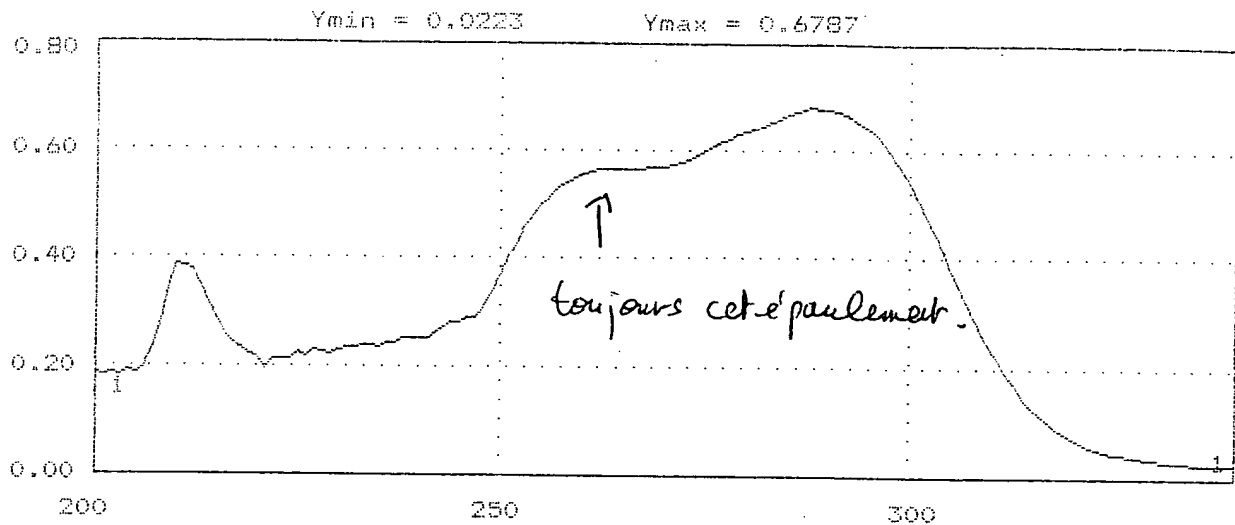
FOURNIER 1001505  
Highly Confidential  
Subject to  
Protective Order

026

spectre sur une solution filtrée contre une ligne de base tween 80-21. filth'

SOLUTION A 75 MG/L FEND - LIGNE DE BASE TWEEN FILTRE/TWEEN FILTRE

25-02-1996 10:55



CONTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001506

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Protective Order

26/02/97

Dissolution dans le  
tween 80 . 27 .

75TPN

02

Comprimés lot 340 .

1. Manière première .

comprimés de Fénofibrate 100 mg .

lot # 340 . date 02/07/97 . de Pharma Pass .

2. Préparation du milieu de dissolution .

voir page 017

3. Pesée des bols de dissolution .

pesée sur PC16 GAL III de 1l de solution de tween à 27  
soit une pesée de : 1001,6 g . (387 à d=1 et 17 à d=10)

bol	remise à zéro	pesée	signature
1	oui	1001,6 g.	M 26.02.97
2	oui	1001,6 g	M 26.02.97
3	oui	1001,6 g.	M 26.02.97
4	oui	1001,6 g.	M 26.02.97
5	oui	1001,6 g.	M 26.02.97
6	oui	1001,6 g	M 26.02.97

4. Pesée des comprimés

sur balance AG 204 GAL 205 .

FOURNIER 1001507  
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Protective Order



028

26.02.97

Code

13:35:28

340

26.02.97

Code

13:38:35

340

ID

1

Code

4

0.0000 g

463.0 mg

0.0 mg

462.8 mg

ID

2

Code

5

0.0 mg

453.5 mg

0.0 mg

461.3 mg

ID

3

Code

6

0.0 mg

478.2 mg

0.0 mg

460.2 mg

5. Conditions de dissolution

dissolutes GAL 103

spallettas 75 TPM.  $\Sigma$  26/04/92milieu  $37 \pm 0,5^\circ \text{C}$   $\Sigma$  26/04/92

spectro 330 GAL 108 avec cuves de 2 mm.

 $\lambda = 290 \text{ nm}$ .

prélèvement avec seringues de 10 ml : prélèvement de  
5 ml de milieu et remplacement par 5 ml de milieu "neuf".  
les seringues sont munies de filtres Prolabo ref 178-3925-01

les lectures se font après filtration sur filtre Millex SLHA025NB.

Rq: le témoin 80 à 2% dans la cuve référence est filtré.

FOURNIER 1001508

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Subject to  
Protective Order

6. Regress

COMPRIMES DE FENDOFIBRATE A 100 MG PHARMA PASS LOT 340 TWEEN 80 2 POUR 100

26-02-1996 15:20

Lambda	No.	Valeur_E
290.0	1	-0.0001_1
290.0	2	-0.0025_1
290.0	3	-0.0051_1
290.0	4	-0.0029_1 <i>T0</i>
290.0	5	-0.0017_1
290.0	6	-0.0031_1
290.0	7	0.5566_1
290.0	8	0.6396_1
290.0	9	0.5303_1 <i>T10</i>
290.0	10	0.5834_1
290.0	11	0.5723_1
290.0	12	0.6159_1
290.0	13	0.7847_1
290.0	14	0.7631_1
290.0	15	0.6748_1 <i>T20</i>
290.0	16	0.6788_1
290.0	17	0.6302_1
290.0	18	0.6067_1
290.0	19	0.8341_1
290.0	20	0.8402_1
290.0	21	0.8585_1 <i>T30</i>
290.0	22	0.7792_1
290.0	23	0.8561_1
290.0	24	0.8434_1
290.0	25	0.8635_1
290.0	26	0.8522_1
290.0	27	0.8721_1 <i>T40</i>
290.0	28	0.8627_1
290.0	29	0.8556_1
290.0	30	0.8471_1

CONTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001509

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le 13/3/97

Dissolution comprimé Pharma Pan  
de 100mg de fénofibrate  
lot 361,

1. Préparation du milieu de dissolution LSWc 0,025M.

masse molaire du LSWc = 289,4 g.

pesée de l'eau : tare = 2,775 kg AC 13/3/97, SC 13/03/97  
brut = 20,180 kg AC 13/3/97, SC 13/03/97  
net.  $20,180 - 2,775 = 17,405$  kg.

soit  $17,405 \times 0,025 \times 289,4 = 125,5$  g de LSWc à peser.

**PESEE SIMPLE**

Date de la pesée : 1997/03/13 11:20:29



\*ARR1768 \*

PRODUIT => NALAUSEF  
POIDS NET => 0.126 KG  
TARE => 0.266 KG  
POIDS BRUT => 0.392 KG

FOURNIER 1001548

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2. Pesée du milieu de dissolution galien.

1 l de LSWc 0,025M = 100,0g.

RC 03.07.97

bol	guc	Pesée	Signature
1	oui	1001,0g	RC 13.03.97 AG-13/3/97
2	oui	1001,0g	RC 13.03.97 AG-13/3/97
3	oui	1001,0g	RC 13.03.97 AG-13/3/97
4	oui	1001,0g	RC 13.03.97 AG-13/3/97
5	oui	1001,0g	RC 13.03.97 AG-13/3/97
6	oui	1001,0g	RC 13.03.97 AG-13/3/97

### 3. Pesée des comprimés gal. los.

un comprimé de 434 mg contient 100 mg de fénofibrate.

13.03.97	13:30:01				
Code	361		ID		5
ID	1	ID	3		
0.0 mg		0.0 mg		0.0 mg	
434.0 mg		435.4 mg		434.8 mg	
ID	2	ID	4	ID	6
0.0 mg		0.0 mg		0.0 mg	
434.3 mg		433.7 mg		433.5 mg	

FOURNIER 1001549

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### 4. Conditions de dissolution

dissoudre Probas gal. los. à palettes.

$$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$$

$$t = 75 \text{ min}$$

RC 13.03.97

AG-13/3/97

RC 13.03.97

Prelèvement de sal de milieu à l'aide d'une seringue plastique de 10 ml et Plastipack 302188 munie d'un préfiltre et Probas 178 398 sol.

Remplacement du milieu prélevé par sal de milieu neuf.

RC 03.07.97

5. Lecture

sur spectrophotomètre nouvellon 222 gallo? dans cuve de  
2mm de trajet optique.

Filtration des prélèvements par filtres millex HA 0,45µm  
et Millipore SLHA025NB.

IMPRIMES PHARMA PASS 100MB LOT 361

13-03-1996 15:48

Lambda No. Valeur\_E

290.0 1 0.0000\_1

290.0 2 0.0042\_1

290.0 3 0.0001\_1

290.0 4 0.0000\_1

290.0 5 -0.0004\_1

290.0 6 -0.0004\_1

290.0 7 -0.0041\_1

290.0 8 -0.0032\_1

290.0 9 -0.0047\_1

290.0 10 0.2631\_1

290.0 11 0.2456\_1

290.0 12 0.2401\_1

290.0 13 0.3076\_15'

290.0 14 0.2886\_1

290.0 15 0.3149\_1

290.0 16 0.5916\_1

290.0 17 0.5801\_1

290.0 18 0.5813\_16'

290.0 19 0.6464\_1

290.0 20 0.6279\_1

290.0 21 0.6439\_1

290.0 22 0.7378\_1

290.0 23 0.7355\_1

290.0 24 0.7347\_1

290.0 25 0.7517\_15'

290.0 26 0.7515\_1

290.0 27 0.7578\_1

290.0 28 0.7961\_1

290.0 29 0.7904\_1

290.0 30 0.8065\_1

290.0 31 0.8005\_18'

290.0 32 0.8096\_1

290.0 33 0.8136\_1

290.0 34 0.8332\_1

290.0 35 0.8445\_1

290.0 36 0.8480\_1

290.0 37 0.8356\_136'

290.0 38 0.8473\_1

290.0 39 0.8441\_1

A2 air/air

LSNa/LSNa.

A2 LSNa/LSNa.

290.0 40 0.8437\_1

290.0 41 0.8515\_1

290.0 42 0.8532\_1

290.0 43 0.8415\_146'

290.0 44 0.8617\_1

290.0 45 0.8616\_1

290.0 46 0.8566\_1

290.0 47 0.8568\_1

290.0 48 0.8710\_1

290.0 49 0.8500\_136'

290.0 50 0.8694\_1

290.0 51 0.8635\_1

290.0 52 0.8513\_1

290.0 53 0.8615\_1

290.0 54 0.8648\_1

290.0 55 0.8367\_166'

290.0 56 0.8670\_1

290.0 57 0.8660\_1

290.0 58 0.8606\_1

290.0 59 0.8549\_1

290.0 60 0.8650\_1

290.0 61 0.8412\_136'

290.0 62 0.8503\_1

290.0 63 0.8596\_1

290.0 64 0.8613\_1

290.0 65 0.8615\_1

290.0 66 0.8737\_136'

290.0 67 0.8518\_1

290.0 68 0.8645\_1

290.0 69 0.8606\_1

FOURNIER 1001550

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Re03.09.32

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 13/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290nm  
CUVE en mm : 2mm

TITRE : comprimé fénofibrate 100mg PHARMA PASS lot 361  
N° CAHIER : LF 178ter p 64  
FICHIER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 361  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

FOURNIER 1001551

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Subject to  
Protective Order

## PREPARATION DES ECHANTILLONS

masse théorique 434  
dosage théorique 100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	434,00	434,30	435,40	433,70	434,80	433,50
quantité de principe actif	100,00	100,07	100,32	99,93	100,18	99,88

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,263	0,246	0,240	0,308	0,289	0,317
10	1000	0,592	0,580	0,581	0,646	0,628	0,644
15	1000	0,738	0,736	0,735	0,752	0,752	0,758
20	1000	0,796	0,790	0,807	0,801	0,810	0,814
30	1000	0,833	0,848	0,848	0,836	0,847	0,844
40	1000	0,844	0,852	0,853	0,842	0,862	0,862
50	1000	0,857	0,857	0,871	0,850	0,869	0,864
60	1000	0,851	0,862	0,865	0,837	0,867	0,866
90	1000	0,861	0,855	0,865	0,841	0,850	0,860
120	1000	0,861	0,862	0,874	0,852	0,865	0,861

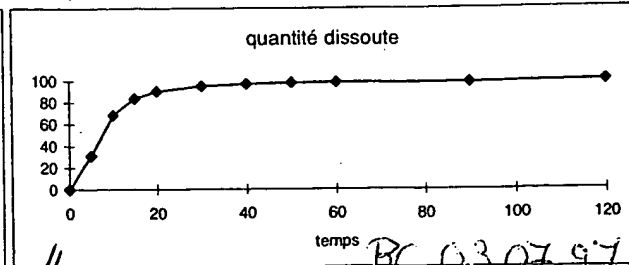
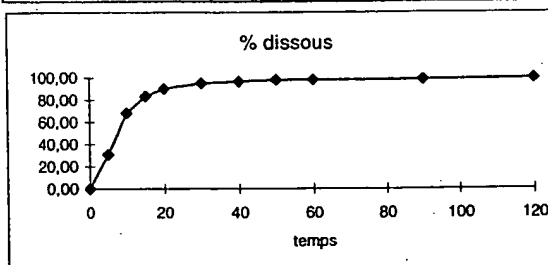
## RESULTATS EN % DISSOUS

*\* Sans incidence significative sur le traitement des données.*

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	30,78	29,22	27,31	26,58	34,25	32,05	35,26	3,63	11,79
10,0	68,09	65,92	64,54	64,48	72,00	69,81	71,81	3,53	5,19
15,0	83,24	82,48	82,18	81,86	84,14	83,91	84,85	1,22	1,47
20,0	90,07	89,33	88,58	90,24	90,01	90,76	91,50	1,03	1,15
30,0	94,88	93,88	95,24	95,23	94,35	95,31	95,29	0,61	0,65
40,0	96,48	95,57	96,38	96,25	95,48	97,45	97,77	0,95	0,98
50,0	97,93	97,48	97,40	98,72	96,84	98,70	98,47	0,80	0,81
60,0	98,04	97,29	98,44	98,53	95,86	98,96	99,17	1,25	1,28
90,0	98,22	98,87	98,14	99,01	96,77	97,56	98,99	0,91	0,93
120,0	99,49	99,35	99,39	100,49	98,46	99,69	99,58	0,65	0,66
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	30,80	29,22	27,33	26,67	34,22	32,11	35,22	3,59	11,67
10,0	68,14	65,92	64,58	64,69	71,95	69,94	71,73	3,47	5,09
15,0	83,29	82,48	82,24	82,12	84,09	84,07	84,76	1,14	1,37
20,0	90,13	89,33	88,65	90,53	89,95	90,93	91,40	1,03	1,14
30,0	94,95	93,88	95,31	95,54	94,28	95,49	95,19	0,69	0,73
40,0	96,54	95,57	96,44	96,56	95,41	97,63	97,65	0,96	1,00
50,0	98,00	97,48	97,47	99,04	96,77	98,88	98,36	0,90	0,92
60,0	98,11	97,29	98,50	98,85	95,80	99,14	99,06	1,32	1,34
90,0	98,29	98,87	98,21	99,33	96,71	97,74	98,87	0,96	0,97
120,0	99,56	99,35	99,46	100,81	98,40	99,87	99,46	0,79	0,79
0,0									
0,0									



RC 03.07.97

Le 14/3/97

## Dissolution comprimés PHARMA PASS

à 100 mg de fénofibrate

Lot 334.

1. Préparation du milieu de dissolution CSA 0,25%.

voir p 64.

2. Pesée du milieu de dissolution gélus.

1 l de CSA 0,25% = 1001,0 g.

bol	gélus	Pesée	Signature.
1	oui	1001,0g	CC 14/03/97 AG 14/3/97
2	oui	1001,0g	CC 14/03/97 AG 14/3/97
3	oui	1001,0g	CC 14/03/97 AG 14/3/97
4	oui	1001,0g	CC 14/03/97 AG 14/3/97
5	oui	<del>oui</del> 1001,0g	CC 14/03/97 AG 14/3/97
6	oui	1001,0g	CC 14/03/97 AG 14/3/97

3. Pesée des comprimés gélus.

un comprimé de 434 mg contient 100 mg de fénofibrate.

FOURNIER 1001552

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RC03.07.97

069

14.03.97

09:23:19

Code

334

ID

٢٣

ID

7.

ID

11

0,0000 g

2,000 g

0.4350 g

0.4401 g

0.4387 g

ID

2

ID

५३

ID

3

0.0000 9

0.0000 g

8,0000 9

0.4386 g

0.4373 g

0.4388 g

#### 4. Conditions de demolition.

demokratisches Problem = palette gel 103.

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$        $\text{Dec-14/3/97}$        $\text{RC 14.03.97}$

8. 75 VPM      88-54/3/27. Re 14.03.97

Prélevement de 5 ml de milieu à l'aide d'une seringue  
de 10 ml sig. Pasteur 202188 munie d'un pipette Probabo  
sig. 178.358.501.

Remplacement du milieu méseux par sol de milieu neuf.

S. Lecture:

sur spectrophotomètre KONTON 822 gal 109 dans cuves de 2mm de trajet optique.

Chaque prélèvement est filtré avant mesure sur Hille  
NA 0,45 µm et Hillepore SLH4025NB.

**FOURNIER 1001553**

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002.07.97



070

OMPRIMES PHARMA PASS LOT 334 100MG FENOFIBRATE

14-03-1996 10:48

Lambda No. Valeur\_E

290.0	1	-0.0000_1	AZ Su low
290.0	2	0.0040_1	LSNa/LSNa
290.0	3	-0.0000_1	AZ LSNa/LSNa
290.0	4	0.3176_1	
290.0	5	0.3109_1	
290.0	6	0.2945_1	
290.0	7	0.2945_1	s'
290.0	8	0.2940_1	
290.0	9	0.2513_1	
290.0	10	0.6466_1	
290.0	11	0.6501_1	
290.0	12	0.6442_1	
290.0	13	0.6524_1	10'
290.0	14	0.6342_1	
290.0	15	0.6020_1	
290.0	16	0.7750_1	
290.0	17	0.7811_1	
290.0	18	0.7670_1	
290.0	19	0.7817_1	15'
290.0	20	0.7736_1	
290.0	21	0.7643_1	
290.0	22	0.8239_1	
290.0	23	0.8215_1	
290.0	24	0.8176_1	
290.0	25	0.8121_1	6'
290.0	26	0.8199_1	
290.0	27	0.8124_1	
290.0	28	0.8691_1	
290.0	29	0.8697_1	
290.0	30	0.8616_1	
290.0	31	0.8632_1	20'
290.0	32	0.8700_1	
290.0	33	0.8683_1	
290.0	34	0.8878_1	
290.0	35	0.8821_1	
290.0	36	0.8925_1	
290.0	37	0.8812_1	40'
290.0	38	0.8803_1	
290.0	39	0.8877_1	
290.0	40	0.8924_1	
290.0	41	0.8911_1	
290.0	42	0.8960_1	
290.0	43	0.8862_1	50'
290.0	44	0.8950_1	
290.0	45	0.8917_1	
290.0	46	0.8923_1	
290.0	47	0.8918_1	
290.0	48	0.9048_1	60'
290.0	49	0.8900_1	
290.0	50	0.8971_1	
290.0	51	0.8997_1	

oude imprimer  
à TO.

==

FOURNIER 1001554  
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BR 03 17 97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 14/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290nm  
CUVE en mm : 2mm

TITRE : comprimé fénofibrate 100mg PHARMA PASS lot 334  
N° CAHIER : LF 178ter p 68  
FICHIER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 334  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 434  
dosage théorique 100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	435,00	438,60	440,10	437,30	438,70	438,80
quantité de principe actif	100,23	101,06	101,41	100,76	101,08	101,11

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,318	0,311	0,295	0,295	0,294	0,251
10	1000	0,647	0,650	0,644	0,652	0,634	0,602
15	1000	0,775	0,781	0,767	0,782	0,774	0,764
20	1000	0,824	0,822	0,818	0,812	0,820	0,812
30	1000	0,869	0,870	0,869	0,863	0,870	0,868
40							
50							
60							

## DISSOLUTION DES COMPRIMES PHARMA PASS A 100 MG DE FENOFIBRATE

### RESULTATS EN % DISSO

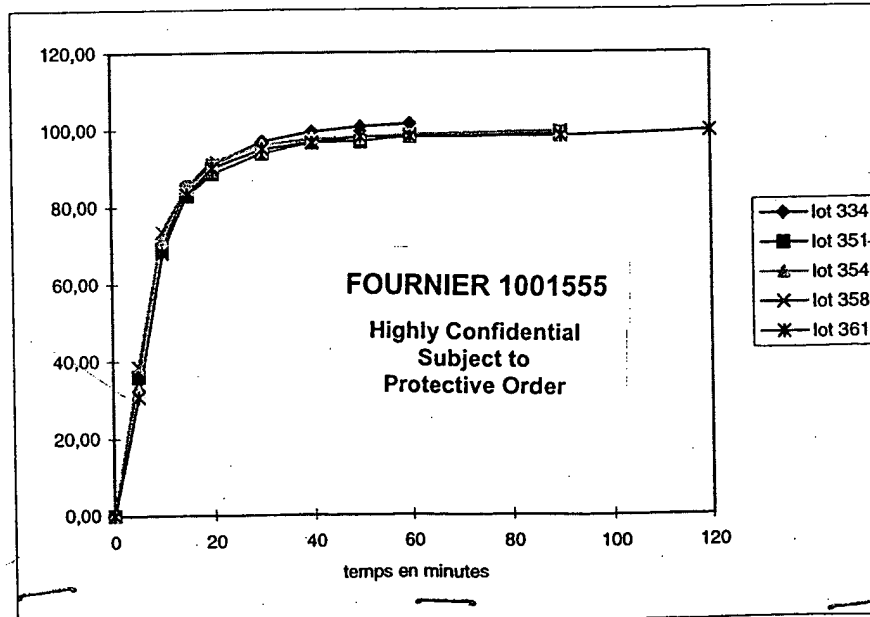
TEMPS
0,0
5,0
10,0
15,0
20,0
30,0
40,0
50,0
60,0
0,0
0,0
0,0
0,0

temps	lot 334	lot 351	lot 354	lot 358	lot 361
0	0,00	0,00	0,00	0,00	0,00
5	32,37	36,09	32,27	38,57	30,78
10	70,41	69,01	71,32	73,54	68,09
15	85,70	82,91	85,82	84,81	83,24
20	90,98	88,55	91,69	91,00	90,07
30	96,83	93,82	95,86	94,10	94,88
40	99,32	96,60	97,47	97,22	96,48
50	100,54	96,74	97,86	97,83	97,93
60	101,47	98,21	98,39	98,58	98,04
90		98,92	98,39	99,39	98,22
120					99,49

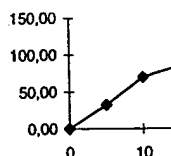
CV
8,12
3,10
1,18
0,86
0,66
0,64
0,42
0,43

### RESULTATS EN QUANTI

TEMPS
0,0
5,0
10,0
15,0
20,0
30,0
40,0
50,0
60,0
0,0
0,0
0,0
0,0



CV
7,93
2,96
0,95
0,63
0,41
0,56
0,38
0,61



AC 03.07.97

AC 03.07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 14/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290nm  
CUVE en mm : 2mm

TITRE : comprimé fénofibrate 100mg PHARMA PASS lot 334  
N° CAHIER : LF 178ter p 68  
FICHIER : m:\commun\glnq\donnbases\lf178ter\dissolution\lot 334  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

**FOURNIER 1001556**  
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Protective Order

## PREPARATION DES ECHANTILLONS

masse théorique 434  
dosage théorique 100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	435,00	438,60	440,10	437,30	438,70	438,80
quantité de principe actif	100,23	101,06	101,41	100,76	101,08	101,11

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

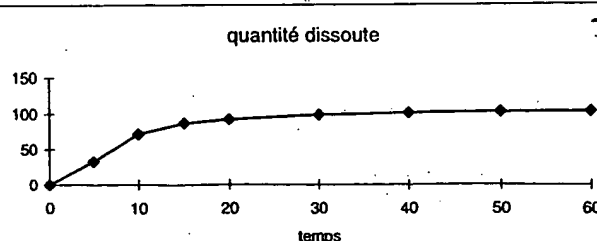
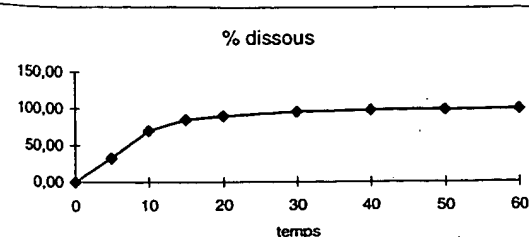
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,318	0,311	0,295	0,295	0,294	0,251
10	1000	0,647	0,650	0,644	0,652	0,634	0,602
15	1000	0,775	0,781	0,767	0,782	0,774	0,764
20	1000	0,824	0,822	0,818	0,812	0,820	0,812
30	1000	0,869	0,870	0,862	0,863	0,870	0,868
40	1000	0,888	0,882	0,893	0,881	0,880	0,888
50	1000	0,892	0,891	0,896	0,886	0,895	0,892
60	1000	0,892	0,892	0,905	0,890	0,897	0,900

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	32,37	35,25	34,19	32,32	32,53	32,32	27,58	2,63	8,12
10,0	70,41	71,90	71,64	70,73	72,06	69,85	66,30	2,18	3,10
15,0	85,70	86,45	86,40	84,56	86,76	85,59	84,43	1,01	1,18
20,0	90,98	92,31	91,33	90,56	90,49	91,07	90,12	0,78	0,86
30,0	96,83	97,75	97,06	95,83	96,57	97,02	96,72	0,64	0,66
40,0	99,32	100,34	98,86	99,70	99,03	98,59	99,40	0,64	0,64
50,0	100,54	101,28	100,33	100,52	100,06	100,73	100,33	0,42	0,42
60,0	101,47	101,77	100,93	102,00	100,99	101,44	101,70	0,43	0,43
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	32,67	35,33	34,56	32,78	32,78	32,67	27,89	2,59	7,93
10,0	71,07	72,07	72,40	71,72	72,61	70,61	67,03	2,10	2,96
15,0	86,50	86,65	87,31	85,74	87,42	86,52	85,36	0,82	0,95
20,0	91,84	92,52	92,30	91,84	91,18	92,06	91,12	0,58	0,63
30,0	97,74	97,98	98,09	97,18	97,30	98,07	97,79	0,40	0,41
40,0	100,25	100,57	99,91	101,10	99,78	99,66	100,50	0,56	0,56
50,0	101,49	101,51	101,40	101,93	100,83	101,82	101,44	0,39	0,38
60,0	102,43	102,01	102,00	103,43	101,76	102,54	102,82	0,63	0,61
0,0									
0,0									
0,0									
0,0									



le 2/3/97.

Le milieu de dissolution choisi pour la suite de l'étude est le LSW à 0,025%.

Dissolution des gélules CANADA  
de Lipidif 200mg micronisé.

Contexte.

dissolutions de  
ces gélules CANADA en vue de la ~~détermination~~ d'étude  
pharmacocinétique.

2 lots à ce jour sont à étudier : lot 48  
lot 49.

A. lot 48.

1 Préparation du LSW à 0,025%.

masse nominale des LSW ~~est~~ 288,4 g.

pesée de l'eau :

tare : 2,805 kg le 2/3/97 RC 20.03.97.

net : 20,220 - 2,805 = 17,425 kg,

net : 20,220 kg le 2/3/97 RC 20.03.97

soit  $17,425 \times 0,025 \times 288,4 = 125,6g$  de LSW à peser.

FOURNIER 1001557

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RC 03.07.97

## PESEE SIMPLE

Date de la pesée : 1997/03/20 11:21:05



\*ARR1768 \*

PRODUIT => NALAUSF  
 POIDS NET => 0.126 KG  
 TARE => 0.262 KG  
 POIDS BRUT => 0.388 KG

//

2. Pesée du milieu de dissolution gélule.

1 litre de LNA 0,025M = 100,0 g.

boîtes	gélule	Pesée	Signature.
1	oui	1001,0g	RC 20.03.97. AR-20/3/97
2	oui	1001,0g	RC 20.03.97 AR-20/3/97
3	oui	1001,0g	RC 20.03.97 AR-20/3/97
4	oui	1001,0g	RC 20.03.97 AR-20/3/97
5	oui	1001,0g	RC 20.03.97 AR-20/3/97
6	oui	1001,0g	RC 20.03.97 AR-20/3/97

3. Conditions de dissolution.

dissolvent à palettes gél 103.

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T° = 37°C ± 0,5°C AR-20/3/97. RC 20.03.97

S: 75 rpm AR-20/3/97 RC 20.03.97

Prélèvement de 5 ml de milieu à T: 5, 10, 15, 20, 30, 40, 50, 60,  
 120 min avec une seringue plastique de 20 ml ref.

Plastipack, 302188 munie d'un préfiltre. Protéger ref. 178398 soit  
 Remplacement du milieu prélevé par 5 ml de milieu neuf.

RC 03.07.97

#### 4. observations

sur spectrophotométrie nouveau 320 gel 103

dans cuve de 2 mm de trajet optique.

filtration des échantillons sur Millipore 0,45 µm HA GF/A625NB

LULES LIP 200M CANADA LOT 48

20-03-1996 15:47

Lambda	No.	Valeur_E			
290.0	1	0.0000_1	A2 au/au		
290.0	2	0.0025_1	LSNa/LSNa.		
290.0	3	0.0001_1	A2 LSNa/LSNa.		
290.0	4	0.0004_1			
290.0	5	-0.0025_1		290.0	34 1.2040_1
290.0	6	-0.0004_1		290.0	35 1.1491_1
290.0	7	-0.0004_1		290.0	36 1.1749_1
290.0	8	-0.0001_1		290.0	37 1.1827_1
290.0	9	0.0028_1		290.0	38 1.2034_1
290.0	10	0.0671_1		290.0	39 1.1166_1
290.0	11	0.0245_1		290.0	40 1.2901_1
290.0	12	0.0894_1		290.0	41 1.2844_1
290.0	13	0.0672_1		290.0	42 1.2915_1
290.0	14	0.0754_1		290.0	43 1.2818_1
290.0	15	0.0434_1		290.0	44 1.3081_1
290.0	16	0.4295_1		290.0	45 1.2346_1
290.0	17	0.2436_1		290.0	46 1.3703_1
290.0	18	0.3844_1		290.0	47 1.3504_1
290.0	19	0.4117_1		290.0	48 1.3473_1
290.0	20	0.3846_1		290.0	49 1.3515_1
290.0	21	0.2100_1		290.0	50 1.3676_1
290.0	22	0.7951_1		290.0	51 1.2904_1
290.0	23	0.6496_1		290.0	52 1.4049_1
290.0	24	0.7340_1		290.0	53 1.3748_1
290.0	25	0.7582_1		290.0	54 1.3742_1
290.0	26	0.7614_1		290.0	55 1.3709_1
290.0	27	0.6386_1		290.0	56 1.4313_1
290.0	28	0.9769_1		290.0	57 1.3416_1
290.0	29	0.8869_1		290.0	58 1.5295_1
290.0	30	0.9400_1		290.0	59 1.5386_1
290.0	31	0.9543_1		290.0	60 1.5361_1
290.0	32	0.9794_1		290.0	61 1.5358_1
290.0	33	0.8447_1		290.0	62 1.5554_1
				290.0	63 1.4657_1

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RC 03.07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 20/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lip200 M Canada lot 48  
N° CAHIER : Lf 178ter dissolution n°1 p72  
FICHIER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 48 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

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## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoïn 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

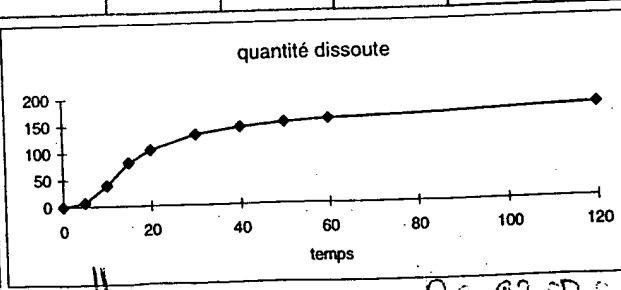
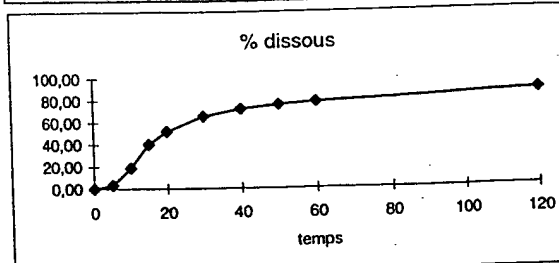
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,067	0,025	0,089	0,067	0,075	0,043
10	1000	0,43	0,244	0,384	0,412	0,385	0,21
15	1000	0,795	0,65	0,734	0,758	0,761	0,639
20	1000	0,977	0,887	0,94	0,954	0,979	0,845
30	1000	1,204	1,149	1,175	1,183	1,203	1,117
40	1000	1,29	1,284	1,292	1,282	1,308	1,235
50	1000	1,37	1,35	1,347	1,352	1,368	1,29
60	1000	1,405	1,375	1,374	1,371	1,431	1,342
120	1000	1,53	1,539	1,536	1,536	1,555	1,466

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0	1,28	37,90
5,0	3,39	3,72	1,39	4,94	3,72	4,17	2,39	5,17	27,03
10,0	19,14	23,91	13,56	21,36	22,91	21,41	11,68	3,58	8,88
15,0	40,27	44,30	36,19	40,91	42,24	42,41	35,57	3,02	5,81
20,0	52,00	54,64	49,53	52,56	53,34	54,73	47,19	1,93	2,94
30,0	65,67	67,52	64,34	65,87	66,33	67,44	62,54	1,43	1,98
40,0	72,11	72,63	72,15	72,70	72,16	73,61	69,40	1,68	2,22
50,0	76,04	77,43	76,18	76,12	76,40	77,31	72,80	1,78	2,27
60,0	78,46	79,76	77,94	77,99	77,84	81,19	76,05	1,80	2,07
120,0	86,84	87,09	87,43	87,37	87,38	88,48	83,31		
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0	2,57	37,90
5,0	6,78	7,44	2,78	9,89	7,44	8,33	4,78	10,35	27,03
10,0	38,27	47,82	27,13	42,72	45,82	42,82	23,36	7,15	8,88
15,0	80,54	88,61	72,37	81,82	84,49	84,81	71,14	6,05	5,81
20,0	104,00	109,27	99,07	105,12	106,69	109,46	94,38	3,86	2,94
30,0	131,35	135,04	128,67	131,75	132,66	134,89	125,08	2,86	1,98
40,0	144,22	145,26	144,31	145,40	144,32	147,22	138,81	3,37	2,22
50,0	152,08	154,87	152,36	152,23	152,81	154,62	145,61	3,56	2,27
60,0	156,92	159,52	155,88	155,98	155,67	162,38	152,10	3,59	2,07
120,0	173,69	174,19	174,87	174,74	174,77	176,95	166,62		
0,0									
0,0									
0,0									



RC 03.07.97

21/3/97

B. lot 49.1. Préparation du milieu de dissolution LSNa 90254.

voir p 72.

2. Pesée du milieu de dissolution galen.

1 l de LSNa = 1001,0 g.

bol	Remise à jour	Pesée	Signature.
1	oui	1001,0g	RC 29.03.97 AB 21/03/97
2	oui	1001,0g	RC 21.03.97 AB 21/03/97
3	oui	1001,0g	RC 21.03.97 AB 21/03/97
4	oui	1001,0g	RC 21.03.97 AB 21/03/97
5	oui	1001,0g	RC 21.03.97 AB 21/03/97
6	oui	1001,0g	RC 21.03.97 AB 21/03/97

3. Conditions de dissolution

dissolvant Triolabs galen à palettes tournantes.

 $T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$ . AB 21/3/97 RC 21.03.97

5: 75 RPM AB 21/3/97 RC 21.03.97

Prélèvement de S.N à  $T = 5, 10, 15, 20, 30, 40, 50, 60, 120$  min  
à l'aide d'une seringue plastique de 10 ml <sup>ref</sup> Plastipack  
Zal 188 munie d'un préfiltre Triolabs <sup>ref</sup>: 178398 sol.

Remplacement du milieu prélevé par S.N de milieu neuf.

FOURNIER 1001561  
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RC 03.07.97



4. Thèmes**DISSOLUTION**

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

**CONDITIONS ET MODES OPERATOIRES**

OPERATEUR : A GRANDJEAN  
DATE : 21/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lip200 M Canada lot 49  
N° CAHIER : Lf 178ter dissolution n°1 p76  
FICHER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 49 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

**FOURNIER 1001562**

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**PREPARATION DES ECHANTILLONS**

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

**SUIVI DE LA DISSOLUTION**

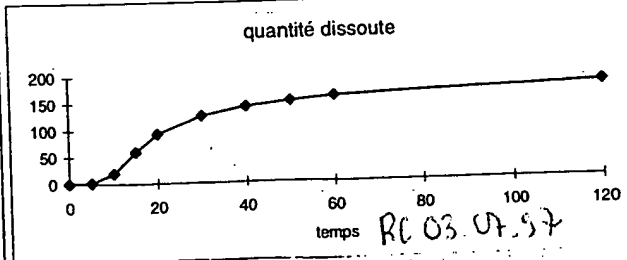
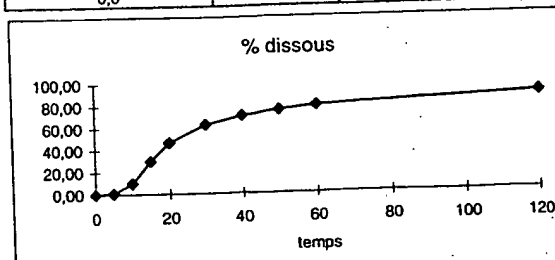
volume prélevé en ml		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
5	1000	0,011	0,012	0,016	0,011	0,018	0,013
10	1000	0,183	0,229	0,127	0,122	0,198	0,201
15	1000	0,526	0,697	0,358	0,509	0,517	0,598
20	1000	0,839	0,946	0,687	0,838	0,818	0,899
30	1000	1,123	1,187	1,071	1,136	1,117	1,125
40	1000	1,237	1,31	1,242	1,259	1,268	1,277
50	1000	1,34	1,351	1,33	1,34	1,365	1,357
60	1000	1,382	1,415	1,412	1,387	1,433	1,416
120	1000	1,532	1,577	1,577	1,543	1,578	1,54

**RESULTATS EN % DISSOUS**

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	1,00	0,72	0,16	21,34
5,0	0,75	0,61	0,67	0,89	0,61	11,01	11,17	2,39	24,38
10,0	9,82	10,17	12,73	7,06	6,78	33,28	33,28	6,23	20,94
15,0	29,73	29,28	38,79	19,93	28,31	45,65	50,17	4,92	10,52
20,0	46,75	46,81	52,82	38,31	46,73	62,49	62,98	2,13	3,38
30,0	63,02	62,82	66,47	59,83	63,52	71,19	71,73	1,54	2,16
40,0	71,05	69,47	73,63	69,63	70,67	76,93	76,53	0,77	1,01
50,0	75,94	75,53	76,27	74,86	75,52	81,08	80,19	1,09	1,37
60,0	79,67	78,24	80,20	79,79	78,50	89,54	87,47	1,21	1,37
120,0	88,41	86,96	89,60	89,35	87,56				
0,0									
0,0									
0,0									

**RESULTATS EN QUANTITE DISSOUE**

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	2,00	1,44	0,32	21,34
5,0	1,50	1,22	1,33	1,78	1,22	22,01	22,34	4,79	24,38
10,0	19,64	20,34	25,45	14,12	13,56	57,56	66,56	12,45	20,94
15,0	59,46	58,55	77,58	39,86	56,63	91,30	100,34	9,84	10,52
20,0	93,50	93,62	105,63	76,61	93,47	124,97	125,95	4,26	3,38
30,0	126,03	125,64	132,94	119,66	127,04	142,37	143,46	3,07	2,16
40,0	142,10	138,93	147,26	139,26	141,34	153,85	153,06	1,53	1,01
50,0	151,88	151,07	152,55	149,72	151,04	162,17	160,37	2,19	1,37
60,0	159,33	156,48	160,41	159,57	157,01	179,07	174,94	2,41	1,37
120,0	176,82	173,91	179,19	178,69	175,11				
0,0									
0,0									
0,0									



## 4. Mesures

sur spectrophotomètre KONTRON 230 gel 109 dans cuves de 2mm de  
trajet optique.

LONGUEUR D ONDE FIXE

21-03-1996 15:03

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0025_1
290.0	3	-0.0001_1
290.0	4	-0.0001_1
290.0	5	0.0004_1
290.0	6	0.0002_1
290.0	7	-0.0014_1
290.0	8	0.0002_1
290.0	9	0.0002_1
290.0	10	0.0108_1
290.0	11	0.0123_1
290.0	12	0.0157_1
290.0	13	0.0107_1
290.0	14	0.0300_1
290.0	15	0.0180_1
290.0	16	0.0129_1
290.0	17	0.1833_1
290.0	18	0.2292_1
290.0	19	0.1267_1
290.0	20	0.1219_1
290.0	21	0.1975_1
290.0	22	0.2010_1
290.0	23	0.5260_1
290.0	24	0.6972_1
290.0	25	0.3578_1
290.0	26	0.5086_1
290.0	27	0.5166_1
290.0	28	0.5984_1
290.0	29	0.8387_1
290.0	30	0.9457_1
290.0	31	0.6873_1
290.0	32	0.8380_1
290.0	33	0.8176_1
290.0	34	0.8989_1
290.0	35	1.1226_1
290.0	36	1.1870_1
290.0	37	1.0706_1
290.0	38	1.1362_1
290.0	39	1.1174_1
290.0	40	1.1251_1
290.0	41	1.2371_1
290.0	42	1.3103_1
290.0	43	1.2421_1
290.0	44	1.2589_1
290.0	45	1.2682_1
290.0	46	1.2769_1
290.0	47	1.3400_1
290.0	48	1.3511_1
290.0	49	1.3300_1
290.0	50	1.3398_1
290.0	51	1.3651_1
290.0	52	1.3573_1
290.0	53	1.3821_1
290.0	54	1.4146_1
290.0	55	1.4116_1
290.0	56	1.3867_1
290.0	57	1.4327_1
290.0	58	1.4159_1
290.0	59	1.5316_1
290.0	60	1.5774_1
290.0	61	1.5772_1
290.0	62	1.5433_1
290.0	63	1.5783_1
290.0	64	1.5404_1

AZ au laui  
LSNa / LSNa -  
AZ LSNa / LSNa.

une goutte coule à l'essai.  
de la cuve  $\Rightarrow$  Bettern refait.

FOURNIER 1001563

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suite de l'étude p 22.

Le 24/3/97

C. Lot 50

1. Préparation du milieu de dissolution LSNa 0,025M  
masse molaire du LSNa = 283,4 g.

pesée de l'eau: Tare 2,830 kg. le 24/3/97. M 24.03.97  
luer 17,960 kg. le 24/3/97. se 24/03/97  
net : 17,960 - 2,830 = 15,090 kg.

soit  $15,090 \times 283,4 \times 0,025 = 108,8 \text{ g}$  de LSNa à peser,

**PESEE SIMPLE**

Date de la pesée : 1997/03/24 10:44:12



\*ARR1768 \*

PRODUIT => NALAUSF  
POIDS NET => 0.109 KG  
TARE => 0.265 KG  
POIDS BRUT => 0.374 KG

FOURNIER 1001564  
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Subject to  
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//

2. Pesée du milieu de dissolution galu.

1 l de LSNa 0,025M = 1001,0 g.

bol	gus	Pesée	Signatures
1	oui	1001,0g	se 24/03/97 le 24/3/97
2	oui	1001,0g	se 24/03/97 le 24/3/97
3	oui	1001,0g	se 24/03/97 le 24/3/97
4	oui	1001,0g	se 24/03/97 le 24/3/97
5	oui	1001,0g	se 24/03/97 le 24/3/97
6	oui	1001,0g	se 24/03/97 le 24/3/97

RC.03.09.97

### 3. Conditions de dissolution

dissolvant Probas gal 103 équipé de palettes tournantes.

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  AR-24/3/97. M 24.03.47

$\gamma: 75 \text{ RPM}$  AR-24/3/97 M 24.03.97

Prélèvement de 5 ml de milieu à  $T = 5, 10, 15, 20, 30, 40, 50, 60, 120 \text{ min}$

avec une seringue plastique de 10 ml réf: Plastipack 302188

munie d'un préfiltre Probas réf: 178398501.

Remplacement du milieu prélevé par 5 ml de milieu neuf.

### 4. Recherches

sur spectrophotomètres KONTRON 930 gal 103.

dans cuves de 2 mm de trajet optique.

filtration des échantillons sur filtres Miller NA 045 sur membrane

SCHAEFEL.

LONGUEUR D ONDE FIXE

24-03-1996 15:24

Lambda	No.	Valeur_E
290.0	1	-0.0000_1
290.0	2	-0.0006_1
290.0	3	0.0001_1
290.0	4	-0.0003_1
290.0	5	-0.0020_1
290.0	6	-0.0020_1
290.0	7	-0.0002_1
290.0	8	-0.0002_1
290.0	9	-0.0002_1
290.0	10	0.0482_1
290.0	11	0.0151_1
290.0	12	0.0638_1
290.0	13	0.0296_1
290.0	14	0.0122_1
290.0	15	0.0223_1
290.0	16	0.2303_1
290.0	17	0.1394_1
290.0	18	0.4416_1
290.0	19	0.1933_1
290.0	20	0.2776_1
290.0	21	0.1737_1

AR au / au  
=  $\frac{A_{Na}}{A_{Na}}$   
AR  $\frac{A_{Na}}{A_{Na}}$

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R 03 07 97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 24/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lip200 M Canada lot 50  
N° CAHIER : Lf 178ter dissolution n°1 p78  
FICHIER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 50 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

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## SUIVI DE LA DISSOLUTION

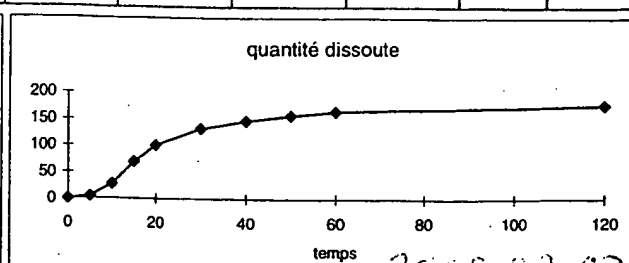
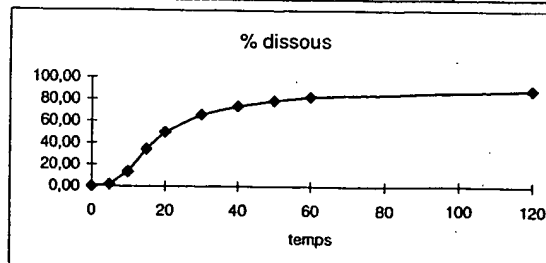
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,048	0,015	0,064	0,03	0,012	0,022
10	1000	0,23	0,139	0,442	0,193	0,278	0,174
15	1000	0,531	0,473	0,839	0,562	0,734	0,567
20	1000	0,842	0,789	1,028	0,867	0,977	0,876
30	1000	1,158	1,117	1,244	1,159	1,23	1,168
40	1000	1,3	1,296	1,323	1,299	1,335	1,308
50	1000	1,39	1,382	1,43	1,364	1,406	1,377
60	1000	1,437	1,443	1,476	1,451	1,466	1,443
120	1000	1,587	1,545	1,564	1,497	1,592	1,541

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	1,77	2,67	0,83	3,56	1,67	0,67	1,22	1,13	63,96
10,0	13,49	12,79	7,73	24,57	10,73	15,45	9,67	6,04	44,79
15,0	34,39	29,58	26,32	46,75	31,28	40,86	31,55	7,75	22,55
20,0	50,05	47,00	44,01	57,48	48,38	54,56	48,88	5,01	10,01
30,0	66,02	64,79	62,45	69,77	64,85	68,89	65,34	2,77	4,20
40,0	73,61	73,00	72,70	74,50	72,95	75,06	73,45	0,96	1,30
50,0	78,49	78,36	77,84	80,82	76,92	79,38	77,64	1,40	1,79
60,0	82,28	81,36	81,61	83,77	82,13	83,10	81,69	0,95	1,16
120,0	88,33	90,09	87,68	89,07	85,09	90,51	87,54	2,00	2,26
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	3,54	5,33	1,67	7,11	3,33	1,33	2,44	2,26	63,96
10,0	26,98	25,58	15,45	49,15	21,46	30,90	19,35	12,09	44,79
15,0	68,78	59,15	52,64	93,50	62,57	81,72	63,11	15,51	22,55
20,0	100,11	94,01	88,02	114,97	96,77	109,12	97,76	10,02	10,01
30,0	132,03	129,58	124,90	139,54	129,70	137,78	130,69	5,54	4,20
40,0	147,22	146,01	145,41	149,01	145,90	150,13	146,89	1,91	1,30
50,0	156,99	156,73	155,68	161,63	153,84	158,76	155,29	2,80	1,79
60,0	164,56	162,72	163,23	167,54	164,26	166,21	163,38	1,91	1,16
120,0	176,66	180,19	175,36	178,14	170,18	181,02	175,08	4,00	2,26
0,0									
0,0									
0,0									



RC03.07.97

le 25/3/97. u

Dissolution lipanthyl loc H

lot 2177.

contexte: comparer cette dissolution avec celles des gâtelles lipanthylabo  
lots CANADA.

1. Préparation du LSNa 0,025M.

voir p78.

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2. Pesée du milieu de dissolution gâtelles.

1 L de LSNa 0,025M = 1001,0g.

lot	gê	Pesée	Signatures.
1	oui	1001,0g	25/03/97 25/3/97
2	oui	1001,0g.	25/03/97 25/3/97
3	oui	1001,0g.	25/03/97 25/3/97
4	oui	1001,0g.	25/03/97 25/3/97
5	oui	1001,0g.	25/03/97 25/3/97
6	oui	1001,0g.	25/03/97 25/3/97.

3. Conditions opératoires

dissolvent à palettes gâtelles.

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  25/3/97 M. 25.03.97

$\gamma = 75 \text{ RPM}$  25/3/97 M. 25.03.97

RC 03.07.97

Prélèvement de Sm<sup>2</sup> de milieu à T = 5, 10, 15, 20, 30, 40, 50, 60, 110 min  
à l'aide d'une seringue plastique 10 ml ref Plastipack 302183  
et munie d'un pistolet PROUSS ref. 17839851

Remplacement du milieu ~~serif~~ prélevé par Sm<sup>2</sup> de milieu neuf.

#### 4. Observes

spectrophotomètre KONTRON 930 gal 107.

dans cuve de 2 mm de trajet optique.

filtrations des échantillons sur filer HA 0,45 µm ref Ritec par  
SCHAULSNER.

BELULES LIP200M LOT 2177

25-03-1996 11:22

Lambda	No.	Valeur_E			
290.0	1	-0.0000_1A2 air/air			
290.0	2	0.0034_1LSNa/LSNa			
290.0	3	0.0001_1A2 LSNa/LSNa			
290.0	4	0.0006_1	290.0	34	1.2158_1
290.0	5	0.0006_1	290.0	35	1.1594_1
290.0	6	0.0010_1b	290.0	36	1.2603_1
290.0	7	-0.0000_1	290.0	37	1.1780_1
290.0	8	0.0040_1	290.0	38	1.2072_1
290.0	9	-0.0001_1	290.0	39	1.2272_1
290.0	10	0.0136_1	290.0	40	1.3287_1
290.0	11	0.0075_1	290.0	41	1.2520_1
290.0	12	0.0291_1	290.0	42	1.3620_1
290.0	13	0.0838_1	290.0	43	1.2580_1
290.0	14	0.0011_1s	290.0	44	1.3143_1
290.0	15	0.0367_1	290.0	45	1.3304_1
290.0	16	0.2963_1	290.0	46	1.3622_1
290.0	17	0.2993_1	290.0	47	1.3102_1
290.0	18	0.3993_1	290.0	48	1.4061_1
290.0	19	0.4682_1b	290.0	49	1.2820_1
290.0	20	0.2434_1	290.0	50	1.3477_1
290.0	21	0.4199_1	290.0	51	1.3777_1
290.0	22	0.7502_1	290.0	52	1.3921_1
290.0	23	0.7177_1	290.0	53	1.3240_1
290.0	24	0.8283_1	290.0	54	1.4211_1
290.0	25	0.8064_1	290.0	55	1.3173_1
290.0	26	0.7137_1s	290.0	56	1.3774_1
290.0	27	0.8476_1	290.0	57	1.4092_1
290.0	28	0.9932_1	290.0	58	1.5022_1
290.0	29	0.9378_1	290.0	59	1.4468_1
290.0	30	1.0423_1	290.0	60	1.5655_1
290.0	31	0.9938_1	290.0	61	1.4417_1
290.0	32	0.9557_1	290.0	62	1.5293_1
290.0	33	1.0399_1	290.0	63	1.5311_1

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RC 03.09.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 25/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lip200 M lot 2177  
N° CAHIER : LJ 178ter dissolution n°1 p81  
FICHIER : m:\commun\glnq\donnbase\l178ter\dissolution\lot 2177  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0.900

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## SUIVI DE LA DISSOLUTION

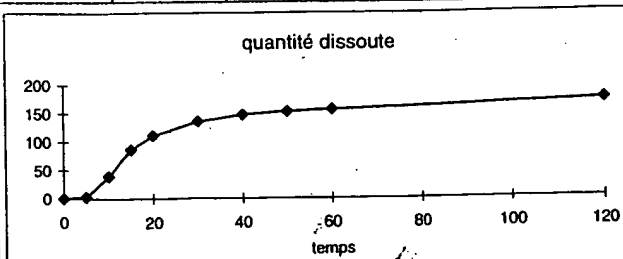
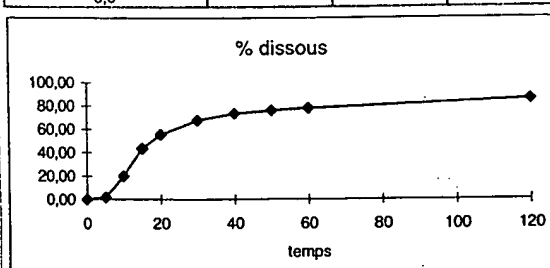
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,014	0,008	0,029	0,084	0,001	0,037
10	1000	0,296	0,299	0,399	0,468	0,243	0,42
15	1000	0,75	0,718	0,828	0,806	0,714	0,848
20	1000	0,993	0,938	1,042	0,994	0,956	1,04
30	1000	1,216	1,159	1,26	1,178	1,207	1,227
40	1000	1,329	1,252	1,362	1,258	1,314	1,33
50	1000	1,362	1,31	1,406	1,282	1,348	1,378
60	1000	1,392	1,324	1,421	1,317	1,377	1,409
120	1000	1,502	1,447	1,566	1,442	1,529	1,531

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	1,60	0,78	0,44	1,61	4,67	0,06	2,06	1,67	104,49
10,0	19,68	16,45	16,61	22,17	26,02	13,50	23,34	4,86	24,67
15,0	43,29	41,75	39,97	46,12	44,93	39,73	47,24	3,23	7,47
20,0	55,54	55,46	52,40	58,24	55,60	53,38	58,14	2,39	4,31
30,0	67,70	68,13	64,93	70,64	66,10	67,59	68,82	2,01	2,98
40,0	73,57	74,74	70,42	76,66	70,87	73,87	74,88	2,45	3,32
50,0	76,17	76,94	73,99	79,48	72,55	76,12	77,92	2,55	3,34
60,0	77,97	78,99	75,13	80,70	74,85	78,11	80,02	2,47	3,17
120,0	85,54	85,49	82,34	89,15	82,16	86,93	87,19	2,81	3,28
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	3,20	1,56	0,89	3,22	9,33	0,11	4,11	3,35	104,49
10,0	39,37	32,90	33,23	44,35	52,05	27,00	46,69	9,71	24,67
15,0	86,58	83,51	79,95	92,24	89,86	79,47	94,48	6,47	7,47
20,0	111,07	110,92	104,79	116,48	111,20	106,75	116,28	4,78	4,31
30,0	135,40	136,25	129,87	141,28	132,20	135,17	137,64	4,03	2,98
40,0	147,15	149,48	140,85	153,31	141,74	147,73	149,76	4,89	3,32
50,0	152,33	153,89	147,99	158,96	145,10	152,24	155,83	5,09	3,34
60,0	155,94	157,98	150,27	161,40	149,71	156,21	160,04	4,94	3,17
120,0	171,09	170,97	164,67	178,30	164,33	173,87	174,38	5,61	3,28
0,0									
0,0									
0,0									

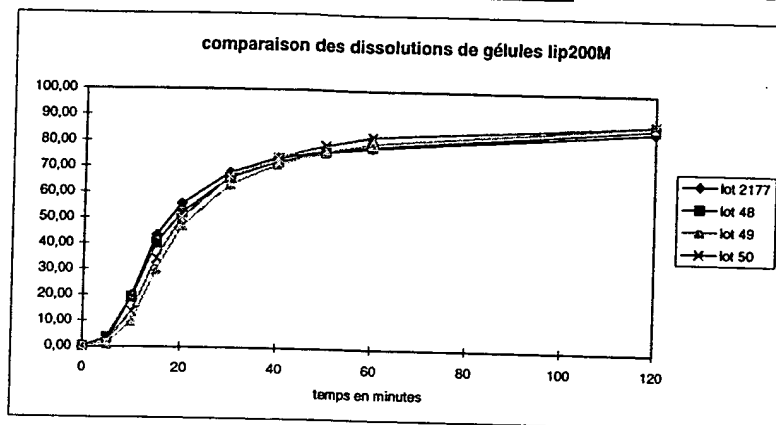


RC 0307 97



temps	lots Canada			
	lot 2177	lot 48	lot 49	lot 50
0	0,00	0,00	0,00	0,00
5	1,60	3,39	0,75	1,77
10	19,68	19,14	9,82	13,49
15	43,29	40,27	29,73	34,39
20	55,54	52,00	46,75	50,05
30	67,70	65,67	63,02	66,02
40	73,57	72,11	71,05	73,61
50	76,17	76,04	75,94	78,49
60	77,97	78,46	79,67	82,28
120	85,54	86,84	88,41	88,33

données de base cahier LI178ter dissolution n°1  
pages 72 à 83



Le lot 2177 n'est pas différent des lots canadiens, il faut noter  
tout de même l'écart à 10 min, 15 min et 30 min d'émulsion 10%.

RC 03 07 97

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17

le 3/4/97

085

Dissolution lot 2394/01 RG.

deux doses ont été restées = 14 et 18 kg.

A. Dose 14 kg.

1. Préparation du LSC 0,025H.

masse nominale du LSC = 288,4 g.

pesée eau Tare = 3,080 kg le 3/4/97. le 03/04/98

brut = 20,870 kg le 3/4/97. le 03/04/98

net = 20,870 - 3,080 = 17,790 kg.

net 17,790 x 0,025 x 288,4 = 128,3 g.

# PESEE SIMPLE

Date de la pesée : 1997/04/03 11:26:55



\*ARR1768 \*

PRODUIT => NALAUSF  
POIDS NET => 0.128 KG  
TARE => 0.154 KG  
POIDS BRUT => 0.282 KG

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2. Pesée du milieu de dissolution gél. //

1 L de LSC 0,025H = 1000,0 g.

le 4/4/97

le 03.07.92

bol.	gic	Pesée	Signature,
1	oui	1001,0 g	82 03/04/97 82 3/4/97
2	oui	1001,0 g	82 03/04/97 82 3/4/97
3	oui	1001,0 g	82 03/04/97 82 3/4/97
4	oui	1001,0 g	82 03/04/97 82 3/4/97
5	oui	1001,0 g	82 03/04/97 82 3/4/97
6	oui	1001,0 g	82 03/04/97 82 3/4/97

### 3. Pesée des comprimés gélés.

1 comprimé de 694,4 mg contient 160 mg de fénofibrate.

03.04.97 13:13:14  
Code 2394.01-14

ID 1 0.0000 g  
0.0000 g  
0.6917 g  
ID 2 0.0000 g  
0.0000 g  
0.6970 g  
ID 3 0.0000 g  
0.0000 g  
0.6924 g

ID 4 0.0000 g  
0.6932 g  
ID 5 0.0000 g  
0.6860 g  
ID 6 0.0000 g  
0.6932 g

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### 4. Conditions de dissolution.

dissolvest ProLabo équipé des palettes tournantes gel 103.

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  82 3/4/97 82 03/04/97

$\omega = 120 \text{ rpm}$  82 3/4/97 82 03/04/97

Prélèvement de S N de milieu à  $T = 5, 10, 15, 20, 30, 40, 50, 60, 120 \text{ min}$

03.07.97

à l'aide d'une seringue plastique dont réf Plastipack 302188 UCI  
et munie d'un pifilre Protaso réf 178398501.  
remplacement du milieu prélevé par sel de viten réf.

Dénaturation à refaire. p.90.

vitene 120 ppm

au lieu de 75 ppm.

FOURNIER 1001573

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PC03.07.27

**FOURNIER 1001574**

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RC03.07.97

B Durée 18 kg.

1. Préparation du L<sub>2</sub>Na 0,025M

soit p85.

2. Pesée du milieu de dissolution. galen.

1 l de L<sub>2</sub>Na 0,025M = 1001,0 g.

bol	gène	Pesée	Signature
1	oui	1001,0g	82 04/04/97 AE 4/4/97
2	oui	1001,0g	82 04/04/97 AE 4/4/97
3	oui	1001,0g	82 04/04/97 AE 4/4/97
4	oui	1001,0g	82 04/04/97 AE 4/4/97
5	oui	1001,0g	82 04/04/97 AE 4/4/97
6	oui	1001,0g	82 04/04/97 AE 4/4/97

3. Pesée des comprimés galen.

1 comprimé de 694,4 mg contient 660 mg de fénofibrate.

04.04.97 09:04:40  
Code 2394.01-18

ID	1	ID	3	ID	5
0.0000 g		0.0000 g		0.0000 g	
0.6982 g		0.6978 g		0.6909 g	
ID	2	ID	4	ID	6
0.0000 g		0.0000 g		0.0000 g	
0.6922 g		0.6990 g		0.6945 g	

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4. Conditions opératoires.

dissolver Profaso gal 103 munie des palette raumers.

T° = 37°C ± 0,5°C 82 04/04/97

σ = 12 r 82 04/04/97

AC03.07.97

Durée 14 kg.

suite de la page 95.

## 2. Pesée du milieu de dissolution gal. III.

1 l de LSN 0,025% = 1001,0 g.

bois	jeu	Pesée	Signature
1	oui	1001,0 g	04/04/97 AB 4/4/97
2	oui	1001,0 g	04/04/97 AB 4/4/97
3	oui	sans effet	04/04/97 AB 4/4/97
4	oui	1001,0 g	04/04/97 AB 4/4/97
5	oui	1001,1 g	04/04/97 AB 4/4/97
6	oui	1001,0 g	04/04/97 AB 4/4/97

Pour usage de LSN 0,025% dissolution avec 5 bois.

## 3. Pesée des comprimés gal. I.

un comprimé de 694,4 mg contient 160 mg de fénofibrate.

04.04.97 10:44:05  
Code 2394.01-14

ID		ID	
1	0.0000 g	4	
0.0000 g	0.6930 g		
0.6987 g	ID	5	
2	0.0000 g		
0.0000 g	0.6935 g		
0.6873 g	ID	6	
	0.0000 g		
	0.6833 g		

FOURNIER 1001576  
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RC 03.07.97

# 4. Conditions de dissolution.

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
 DATE : 04/04/97  
 APPAREIL : gal 103 108  
 LONGUEUR D'ONDE : 290nm  
 CUVE en mm: 2mm  
 TITRE : comprimé fénofibrate 160mg lot 2394/01Rg 14kg de dureté.  
 N° CAHIER : LF 178ter p 90  
 FICHIER : m:\commun\glnq\donnbase\178ter\dissolution\lot 2394/01Rg 14kg  
 ELUANT : LSNa 0,025M  
 AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique 694,4  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	698,70	687,30		693,00	693,50	683,30
quantité de principe actif	160,99	158,36	0,00	159,68	159,79	157,44

Témoin 100mg/l 0,900

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### SUIVI DE LA DISSOLUTION

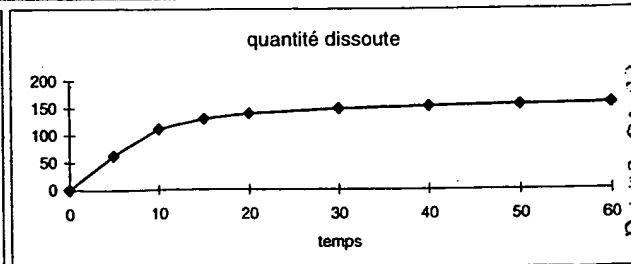
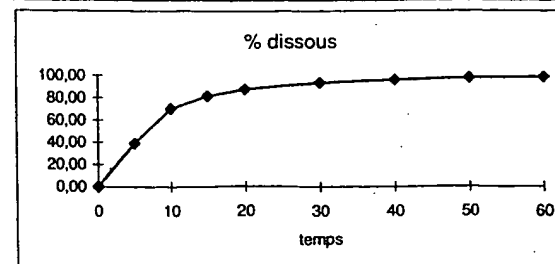
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,498	0,559		0,598	0,511	0,648
10	1000	0,979	0,998		1,027	0,985	1,045
15	1000	1,158	1,160		1,167	1,175	1,173
20	1000	1,245	1,251		1,236	1,264	1,236
30	1000	1,326	1,324		1,307	1,350	1,303
40	1000	1,360	1,359		1,333	1,376	1,331
50	1000	1,379	1,378		1,351	1,399	1,350
60	1000	1,390	1,383		1,355	1,403	1,347

### RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	39,29	34,37	39,22		41,61	35,53	45,73	4,62	11,75
10,0	70,45	67,74	70,22		71,67	68,67	73,98	2,48	3,51
15,0	81,95	80,43	81,93		81,77	82,22	83,38	1,06	1,29
20,0	87,92	86,84	88,73		86,98	88,82	88,24	0,95	1,08
30,0	93,63	92,86	94,29		92,35	95,24	93,40	1,15	1,23
40,0	96,17	95,66	97,21		94,61	97,52	95,84	1,19	1,24
50,0	98,01	97,44	99,02		96,33	99,59	97,65	1,30	1,33
60,0	98,78	98,68	99,85		97,08	100,36	97,91	1,35	1,37
0,0									
0,0									
0,0									
0,0									

### RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	62,53	55,33	62,11		66,44	56,78	72,00	6,89	11,02
10,0	112,18	109,05	111,20		114,44	109,73	116,47	3,17	2,83
15,0	130,49	129,49	129,75		130,57	131,39	131,27	0,86	0,66
20,0	140,01	139,80	140,51		138,88	141,93	138,93	1,27	0,90
30,0	149,10	149,49	149,32		147,46	152,19	147,06	2,04	1,37
40,0	153,15	154,00	153,94		151,08	155,83	150,89	2,12	1,38
50,0	156,08	156,87	156,81		153,82	159,15	153,74	2,30	1,47
60,0	157,30	158,86	158,13		155,01	160,37	154,16	2,63	1,67
0,0									
0,0									
0,0									
0,0									





4. Conditions de dissolution.

dissolvent Probase gal 103 équipé des palettes soudeuses.

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  AC 4/4/97 RC 04.04.97

$\sigma = 75 \text{ RPM}$  AC 4/4/97. RC 04.04.97

Prélèvement de 5 ml de milieu à  $T = 5, 10, 15, 20, 30, 40, 50, 60 \text{ min}$

à l'aide d'une seringue plastique de 10 ml réf Plastipack 302.188

et munie d'un préfiltre Probase réf: 17839850.1.

Remplacement du milieu prélevé par 5 ml de milieu neuf.

5. Analyses.

spectrophotométrie KONTROL 920 gal 108.

cuvette de 2 mm de trajet optique.

filtration des échantillons sur fibres tiller HA réf tillipore 500000 NB

LONGUEUR D ONDE FIXE

04-04-1996 10:51

Lambda	No.	Valeur_E			
290.0	1	0.0000_1	290.0	25	1.2452_1
290.0	2	-0.0051_1	290.0	26	1.2510_1
290.0	3	-0.0001_1	290.0	27	1.2360_1
290.0	4	-0.0001_1	290.0	28	1.2636_1
290.0	5	-0.0001_1	290.0	29	1.2361_1
290.0	6	-0.0001_1	290.0	30	1.3281_1
290.0	7	-0.0001_1	290.0	31	1.3237_1
290.0	8	-0.0001_1	290.0	32	1.3065_1
290.0	9	-0.0001_1	290.0	33	1.3496_1
290.0	10	0.4983_1	290.0	34	1.3031_1
290.0	11	0.5586_1	290.0	35	1.3603_1
290.0	12	0.5983_1	290.0	36	1.3591_1
290.0	13	0.5106_1	290.0	37	1.3331_1
290.0	14	0.6475_1	290.0	38	1.3757_1
290.0	15	0.9794_1	290.0	39	1.3310_1
290.0	16	0.9984_1	290.0	40	1.3789_1
290.0	17	1.0265_1	290.0	41	1.3784_1
290.0	18	0.9845_1	290.0	42	1.3510_1
290.0	19	1.0453_1	290.0	43	1.3989_1
290.0	20	1.1577_1	290.0	44	1.3498_1
290.0	21	1.1595_1	290.0	45	1.3904_1
290.0	22	1.1667_1	290.0	46	1.3829_1
290.0	23	1.1745_1	290.0	47	1.3550_1
290.0	24	1.1727_1	290.0	48	1.4030_1
			290.0	49	1.3469_1

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Le 7/4/97.

Dissolution Compainés E178 (Lot 318 Canada).
enrobés CPADRY OY.
lot 2393/01 RG.

1. Préparation LSNa 0,025M. (CPAD en panne série manuelle),  
masse molaire du LSNa = 283,4 g.

taux eau = 2,725 kg. ~~de~~ 7/4/97 ~~de~~ 07/04/97

mat eau = 28,830 kg ~~de~~ 7/4/97. M 07.04.97

net = 28,830 - 2,725 = 26,105 kg.

soit  $26,105 \times 283,4 \times 0,025 = 188,2$  g. de LSNa

galon. taux = 635,7 g ~~de~~ 7/4/97. M 07.04.97

mat = 824,0 g ~~de~~ 7/4/97 M 07.04.97

net = 824,0 - 635,7 = 188,3 g.

2. Pesée des récipients de dissolution galon.

1 l de LSNa 0,025M = 1001,0 g.

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bol	gros	Pesée	Signature.
1	oui	1001,08.	07/04/97 <del>de</del> 7/4/97
2	oui	1001,08	07/04/97 <del>de</del> 7/4/97
3	oui	1001,08.	07/04/97 <del>de</del> 7/4/97
4	oui	1001,08.	07/04/97 <del>de</del> 7/4/97
5	oui	1001,08	07/04/97 <del>de</del> 7/4/97
6	oui	1001,08	07/04/97 <del>de</del> 7/4/97

08 06/04/97

RC03.07.97

### 3. Essai des comprimés gal 103

00.

Les comprimés contiennent 100 mg de férofenate.

07.04.97 13:40:55  
Code 2393.01

ID	1	ID	3	ID	5
0.0 mg		0.0 mg		0.0 mg	
454.1 mg		460.0 mg		452.4 mg	
ID	2	ID	4	ID	6
0.0 mg		0.0 mg		0.0 mg	
458.9 mg		450.2 mg		448.4 mg	

### 4. Conditions de dissolution.

dissolvant Propaso gal 103 équipé des palettes tournantes.

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  de 7/4/97 M. O. Ch. 97

$t = 75 \text{ rpm}$  de 7/4/97 M. O. Ch. 47

Prélèvement de 5 ml de milieu à  $T = 5, 10, 15, 20, 30, 40, 50, 60 \text{ min}$   
à l'aide d'une seringue plastique de 10 ml ref Plastipack 302183  
munie d'un piston Propaso ref 178398501.

Remplacement du milieu prélevé par 5 ml de milieu neuf.

### 5. Recherche.

spectrophotométrie VIONTRON 920 gal 103.

cuvets de 2 mm de trajet optique.

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LF178TER LOT 2393/01RB ENROBE OPADRY OY

07-04-1996 13:43

Lambda No. Valeur\_E

290.0	1	0.0004_1	02 au/au
290.0	2	-0.0019_1	LS No / LS No
290.0	3	-0.0001_1	02 LS No / LS No.
290.0	4	-0.0031_1	
290.0	5	-0.0035_1	
290.0	6	-0.0048_1	
290.0	7	0.0013_1	
290.0	8	0.0012_1	
290.0	9	0.0014_1	

26/07/97

RC.03.01.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 07/04/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : comprimés enrobés OPADRY OY lot 2393/01RG  
N° CAHIER : LF 178ter dissolution page 92  
FICHIER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 2393rg01  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

FOURNIER 1001581

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## PREPARATION DES ECHANTILLONS

masse théorique	100
dosage théorique	100 en mg

masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
100,00	100,00	100,00	100,00	100,00	100,00
100,00	100,00	100,00	100,00	100,00	100,00

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

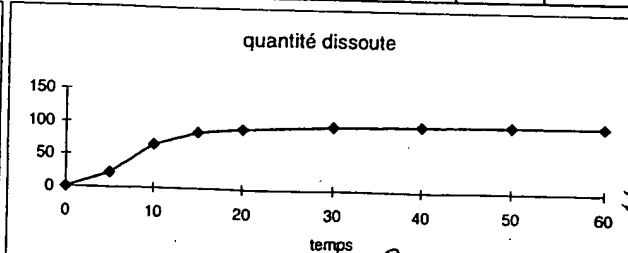
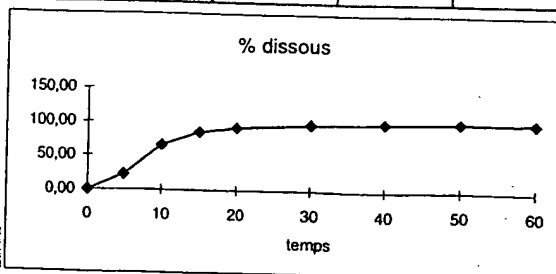
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,164	0,179	0,2	0,212	0,198	0,255
10	1000	0,557	0,565	0,616	0,618	0,6	0,62
15	1000	0,754	0,752	0,773	0,772	0,769	0,776
20	1000	0,825	0,822	0,824	0,817	0,829	0,822
30	1000	0,865	0,876	0,859	0,868	0,873	0,872
40	1000	0,901	0,884	0,883	0,881	0,886	0,886
50	1000	0,906	0,895	0,885	0,882	0,89	0,892
60	1000	0,894	0,893	0,888	0,884	0,893	0,893

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	22,37	18,22	19,89	22,22	23,56	22,00	28,33	3,48	15,55
10,0	66,33	61,98	62,88	68,56	68,78	66,78	69,03	3,14	4,73
15,0	85,55	84,18	83,97	86,34	86,24	85,89	86,71	1,18	1,38
20,0	92,33	92,49	92,16	92,44	91,67	92,98	92,25	0,43	0,47
30,0	97,86	97,39	98,62	96,79	97,79	98,33	98,26	0,68	0,70
40,0	100,35	101,87	100,00	99,93	99,72	100,26	100,30	0,78	0,78
50,0	101,38	102,93	101,71	100,64	100,32	101,20	101,46	0,92	0,91
60,0	101,78	102,10	101,99	101,47	101,03	102,03	102,07	0,44	0,43
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	22,37	18,22	19,89	22,22	23,56	22,00	28,33	3,48	15,55
10,0	66,33	61,98	62,88	68,56	68,78	66,78	69,03	3,14	4,73
15,0	85,55	84,18	83,97	86,34	86,24	85,89	86,71	1,18	1,38
20,0	92,33	92,49	92,16	92,44	91,67	92,98	92,25	0,43	0,47
30,0	97,86	97,39	98,62	96,79	97,79	98,33	98,26	0,68	0,70
40,0	100,35	101,87	100,00	99,93	99,72	100,26	100,30	0,78	0,78
50,0	101,38	102,93	101,71	100,64	100,32	101,20	101,46	0,92	0,91
60,0	101,78	102,10	101,99	101,47	101,03	102,03	102,07	0,44	0,43
0,0									
0,0									
0,0									
0,0									



Re 02.07.97

290.0	10	0.1636_1
290.0	11	0.1793_1
290.0	12	0.2004_1
290.0	13	0.2118_1
290.0	14	0.1981_1
290.0	15	0.2546_1
290.0	16	0.5565_1
290.0	17	0.5649_1
290.0	18	0.6155_1
290.0	19	0.6183_1
290.0	20	0.5997_1
290.0	21	0.6196_1
290.0	22	0.7544_1
290.0	23	0.7523_1
290.0	24	0.7726_1
290.0	25	0.7715_1
290.0	26	0.7686_1
290.0	27	0.7760_1
290.0	28	0.8251_1
290.0	29	0.8218_1
290.0	30	0.8244_1
290.0	31	0.8173_1
290.0	32	0.8287_1
290.0	33	0.8217_1

290.0	34	0.8253_1
290.0	35	0.8764_1
290.0	36	0.8585_1
290.0	37	0.8675_1
290.0	38	0.8734_1
290.0	39	0.8720_1
290.0	40	0.9013_1
290.0	41	0.8842_1
290.0	42	0.8831_1
290.0	43	0.8805_1
290.0	44	0.8858_1
290.0	45	0.8858_1
290.0	46	0.9056_1
290.0	47	0.8954_1
290.0	48	0.8848_1
290.0	49	0.8819_1
290.0	50	0.8897_1
290.0	51	0.8920_1
290.0	52	0.8943_1
290.0	53	0.8930_1
290.0	54	0.8875_1
290.0	55	0.8842_1
290.0	56	0.8930_1
290.0	57	0.8927_1

FOURNIER 1001582

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07.04.97

LOT RG 2394/01

durée 1h45.1. Préparation du milieu de dissolution

Voir page 92

2. Pesée du milieu de dissolution Bal GAL 1111 Ltr de LGNa 0,025N  $\rightarrow$  1001,0 g.

bal	remise à zéro	masse de LGNa 0,025	vérificateurs
1	oui	1001,0 g.	DE 7/4/97. M. 07.04.97
2	oui.	1001,0 g.	DE 7/4/97. M. 07.04.97
3	oui.	1001,0 g.	DE 7/4/97. M. 07.04.97
4	oui.	1001,0 g.	DE 7/4/97. M. 07.04.97
5	oui.	1001,0 g.	DE 7/4/97. M. 07.04.97
6	oui.	1001,0 g.	DE 7/4/97. M. 07.04.97

conditions de dissolution

Dissolutest

T° : 37°C  $\pm$  0,5

DE 7/4/97 M. 07.04.97

GAL 031

Q : 75 TPN

DE 7/4/97 M. 07.04.97

3. Pesée des comprimés Bal GAL 245 M. 07.04.97FOURNIER 1001583  
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n°3 et 5 : Remise à zéro non exacte. Erreur prise en compte pour la valeur nette entrée sur la feuille de calcul. M. 11.07.97

07.04.97 Code 2394.01-14	1	2	3	4	5	6
ID	ID	ID	ID	ID	ID	ID
09:55:45	0.0 mg	0.0 mg	0.1 mg	0.0 mg	0.1 mg	0.0 mg
	688.1 mg	694.3 mg	692.9 mg	695.1 mg	695.4 mg	693.8 mg

M

RC 03.07.97

# 4. Lecture

UV GAL 233 - 290nm - cellule 2mm. chrono GAL 123

LF 178 TER LOT RG 2394/01 14 KG

04-06-1997 22:53

Lambda	No.	Valeur_E
290.0	1	-0.0008_1
290.0	2	-0.0006_1
290.0	3	-0.0001_1
290.0	4	-0.0005_1
290.0	5	0.0007_1
290.0	6	-0.0005_1
290.0	7	0.4756_1
290.0	8	0.3639_1
290.0	9	0.4003_1

290.0	31	1.2746_1
290.0	32	1.2996_1
290.0	33	1.3016_1
290.0	34	1.2448_1
290.0	35	1.2984_1
290.0	36	1.2623_1
290.0	37	1.2993_1
290.0	38	1.3448_1
290.0	39	1.3472_1
290.0	40	1.3248_1
290.0	41	1.3365_1

be due à un problème

ce problème vient de la lecture...? Modifié

ne / 11.07.17

FOURNIER 1001584

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290.0 29 1.1890\_1 290.0 60 1.3949\_1  
290.0 30 1.2406\_1 290.0 60 1.3949\_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

Fournier 1001585

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### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 07/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2  
TITRE : LF 178 TER RG 2394/01 à 14 KG  
N° CAHIER : 178 TER n°1 p.96-3  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2394RG01 14 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

o masses cellule 3 et 5, voir page 45  
M.H. 07 97

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	688,10	694,30	692,80	695,10	695,30	693,80
quantité de principe actif	158,55	159,98	159,63	160,16	160,21	159,86

Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

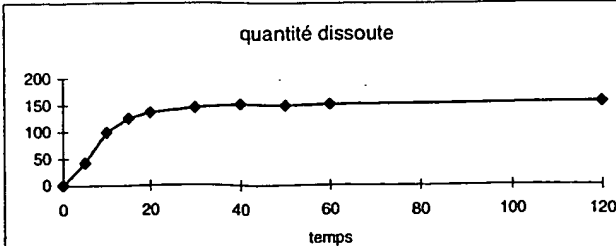
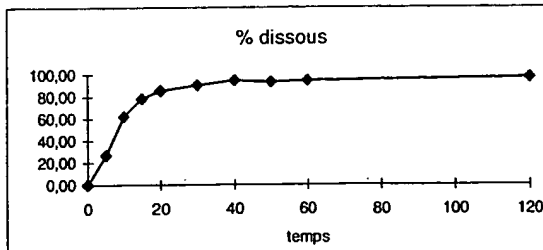
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,476	0,364	0,4	0,347	0,405	0,334
10	1000	0,942	0,811	0,785	0,968	0,916	0,917
15	1000	1,068	1,167	1,116	1,115	1,142	1,097
20	1000	1,214	1,254	1,18	1,198	1,189	1,241
30	1000	1,275	1,3	1,302	1,245	1,298	1,262
40	1000	1,299	1,345	1,347	1,325	1,337	1,351
50	1000	1,321	1,299	1,336	1,266	1,284	1,357
60	1000	1,259	1,291	1,291	1,34	1,366	1,36
120	1000	1,338	1,393	1,375	1,323	1,315	1,395

### RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	26,98	33,36	25,28	27,84	24,07	28,09	23,21	3,69	13,69
10,0	62,04	66,18	56,45	54,78	67,27	63,67	63,85	5,19	8,36
15,0	78,17	75,34	81,46	78,09	77,81	79,66	76,68	2,16	2,77
20,0	85,19	85,95	87,91	82,93	83,95	83,32	87,07	2,08	2,44
30,0	90,32	90,65	91,54	91,84	87,63	91,29	88,96	1,67	1,85
40,0	94,49	92,78	95,12	95,42	93,61	94,44	95,59	1,11	1,18
50,0	93,33	94,77	92,39	95,13	89,98	91,23	96,47	2,52	2,70
60,0	94,68	90,89		92,46	95,55	97,36	97,15	2,89	3,05
120,0	97,36	96,87	99,37	98,75	94,84	94,30	100,06	2,42	2,48
0,0									
0,0									
0,0									

### RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	43,07	52,89	40,44	44,44	38,56	45,00	37,11	5,74	13,33
10,0	99,09	104,93	90,31	87,44	107,75	102,00	102,07	8,24	8,31
15,0	124,88	119,45	130,32	124,66	124,62	127,62	122,58	3,79	3,04
20,0	136,07	136,27	140,63	132,39	134,46	133,48	139,19	3,27	2,40
30,0	144,26	143,72	146,44	146,60	140,35	146,25	142,22	2,61	1,81
40,0	150,94	147,10	152,16	152,32	149,93	151,31	152,81	2,14	1,42
50,0	149,07	150,26	147,80	151,85	144,11	146,16	154,22	3,75	2,52
60,0	151,21	144,11		147,59	153,04	155,98	155,31	5,16	3,41
120,0	155,52	153,59	158,97	157,64	151,89	151,08	159,96	3,81	2,45
0,0									
0,0									
0,0									





697

### 1. Préparation du milieu de dissolution

Vol page 92

1 liter  $\text{LiNa}$  0,025 N  $\rightarrow$  100 mg.

bol	remise à zero	masse LNa 0,025N	vérifications
1	oui	1001,08	2 07/04/92 M. 07.04.97
2	oui	1001,18	82 07/04/92 M. 07.04.97
3	oui	1001,08	2 07/04/92 M. 07.04.97
4	oui	1001,18	2 07/04/92 M. 07.04.97
5	oui	1001,08	2 07/04/92 M. 07.04.97
6	oui	1001,18	2 07/04/92 M. 07.04.97

Dissolve best

7. 37°C ± 0.5

DE 7/4/97 M 07.04.97

GAL 081

75 TPN

AE 7/4/97 M.Oh.47

3. Pesée des comprimés bal GAL 24h  
2.35

205 M. Ct. Oh. 47

**FOURNIER 1001586**

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ID	Code	Time	Weight
1	07.04.97	13:25:16	0.0 mg
2	2394.01-18		692.8 mg
3			0.0 mg
4			692.2 mg
5			0.0 mg
6			694.5 mg
7			0.0 mg
8			695.7 mg
9			0.0 mg
10			683.9 mg
11			0.0 mg
12			685.6 mg

RCOS.09 97

UV GAL 255 - 290 nm - cellule Zimm

LF 178 TER LOT RG 2394/01 18 KG

04-07-1997 02:23

			Lambda	No.	Valeur_E			
1	290.0	1	0.0000	1	290.0	T <sub>0</sub>	1	-0.0003
2	290.0	2	0.0033	2	290.0		2	0.0003
3	290.0	3	0.0001	3	290.0		3	0.0001
				4	290.0		4	0.0001
				5	290.0		5	0.0007
				6	290.0		6	-0.0002
				7	290.0	T <sub>5</sub>	7	0.2513
				8	290.0		8	0.3229
				9	290.0		9	0.2947
				10	290.0		10	0.2100
				31	290.0	T <sub>30</sub>	31	1.2671
				32	290.0		32	1.2328
				33	290.0		33	1.0270
				34	290.0		34	1.2457
				35	290.0		35	1.1814
				36	290.0		36	1.2088
				37	290.0	T <sub>10</sub>	37	1.3128
				38	290.0		38	1.2857
				39	290.0		39	1.2984
				40	290.0		40	1.2194

1 AZ Air / Air

2 LSWer / LSWer

\*  
d  
u  
o  
u  
g

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UV GAL 255



27/02/97

Dissolution dans le  
tween 80 à 24.

75TPN

031

Gelules Lip 200 ARR 1710

FOURNIER 1001511  
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Protective Order

1. Matière première

gelules lipanthyl 200 ARR 1710.

2. Préparation du milieu de dissolution

tween Protebo référence N° lot 2156C  
eau purifiée du jour.

pesée de l'eau :

tare = 2353,3 g CC 27/02/97 AR 27/02/97.

brut = 3754,8 g CC 27/02/97 AR 27/2/97.

net = 6800,3 g.

pesée du tween :

voir le calcul effectué page 17.

pesée à réaliser :  $2(6800,3 + x) = 100x$

$2 \times 6800,3 + 2x = 100x$

$$x = \frac{13601,8}{98}$$

$$x = 138,79$$

pesée à réaliser : 138,79 g soit 149,3 g (138,79 x 1,08)

tare = 3754,8 g CC 27/02/97 AR 27/2/97

remise à zéro : oui AR 27/2/97.

net = 149,3 g CC 27/02/97 AR 27/2/97.

utilisation de la balance PC16 GAL 111.

Agitation avec agitateur à hélice.

### 3. Pesée des gélules

27.02.97 09:56:14 27.02.97 09:58:06  
Code 1710 Code 1710

ID 1	ID 4
0.0000 g	0.0000 g
0.4263 g	0.4258 g
ID 2	ID 5
0.0000 g	0.0000 g
0.4305 g	0.4296 g
ID 3	ID 6
0.0000 g	0.0000 g
0.4310 g	0.4299 g

balance GAL205

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### 4. Pesée des bols de dissolution

pesée sur balance BAC III.

1 l de solution de tween 80 à 2%. soit une pesée de 1001,6 g. (38 l. à d = 1 et 2 l. à d = 1,08)

bol	remise à zéro	pesée	signature
1	oui	1001,6 g	AB-27/2/97.
2	oui	1001,6 g	AB-27/2/97
3	oui.	1001,6 g	AB-27/2/97.
4	oui.	1001,6 g	AB-27/2/97.
5	oui	1001,6 g	AB-27/2/97.
6	oui.	1001,6 g	AB-27/2/97.

## 5. Conditions de dissolutions

033

• dissolutest Prolebo GAL 103

palettes 75 TPD - 27.02.97 M

bain-marie  $37^{\circ}\text{C} \pm 0,5$  27.02.97 M

• UV 330 GAL 108

$\lambda = 290 \text{ nm}$

cuves 2 mm

prélèvement dans les bords avec une seringue de 10 ml  
prélèvement de 5 ml et remplacement avec du  
milieu neuf.

ce prélèvement se fait avec seringue + tuyau à gon  
+ filtres Prolebo ref 178-3985-01.

la lecture de DO se réalise après filtration sur  
filtres Niller SLHA 025 NB.

le tween 80 à 2% dans la cuve référence est  
aussi filtré sur filtre Niller HA.

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034

6. Measures.

GELULES LIP 200 ARR 1710 DANS TWEEN 80 A 2 POUR 100

27-02-1996 11:38

Lambda	No.	Valeur_E	
290.0	1	-0.0042_1	
290.0	2	0.0003_1	
290.0	3	-0.0006_1	
290.0	4	-0.0044_1	T0
290.0	5	0.0002_1	
290.0	6	-0.0084_1	
<hr/>			
290.0	7	0.4413_1	
290.0	8	0.4562_1	
290.0	9	0.4350_1	
290.0	10	0.4512_1	T10
290.0	11	0.4945_1	
290.0	12	0.5060_1	
<hr/>			
290.0	13	0.7999_1	
290.0	14	0.8103_1	
290.0	15	0.7737_1	
290.0	16	0.7836_1	T20
290.0	17	0.8018_1	
290.0	18	0.8273_1	
<hr/>			
290.0	19	0.9833_1	
290.0	20	0.9450_1	
290.0	21	0.9714_1	
290.0	22	0.9430_1	
290.0	23	0.9725_1	T30
290.0	24	1.0118_1	
<hr/>			
290.0	25	1.1225_1	
290.0	26	1.0709_1	
290.0	27	1.0808_1	
290.0	28	1.0592_1	T40
290.0	29	1.0759_1	
290.0	30	1.1085_1	
<hr/>			
290.0	31	1.1773_1	
290.0	32	1.1519_1	
290.0	33	1.1284_1	
290.0	34	1.1386_1	T50
290.0	35	1.1401_1	
290.0	36	1.2030_1	

INTRON INSTRUMENTS

UVIKON 930

FOURNIER 1001514

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Protective Order

036  
26.02.97

- Dissolution LSNa 902M -  
1000 ml - 75 rpm - COMPTIMES

1- Préparation du milieu de dissolution

- Pesée de l'eau purifiée le 26.02.97 - Balance OALOM

Tare = 2,725 Kg RC 26.02.97 ~~26/02/92~~

Brut = 19,550 Kg RC 26.02.97 ~~26/02/92~~

Brut - Tare = Net = 19,550 - 2,725 = 16,825 Kg RC 26.02.97  
~~26/02/92~~

- Pesée de LSNa au 1768 - Balance GALOM

FICHE DE NETTOYAGE		
FOURNIER 1001515	LOCAL N° : <i>salle des Balances</i>	
Highly Confidential Subject to Protective Order		
DATE DE DEBUT D'UTILISATION DU LOCAL : <i>25.02.97</i>		
PRODUIT : <i>Lipanthyl 67 PLACER</i>		
N° DE LOT : <i>DG 153/00</i>		
OPERATION : <i>Pesée A.P.</i>		
DATE DE FIN D'OPERATION : <i>25.02.97</i>		
VIDE DE SALLE EFFECTUÉ PAR : <i>M</i>	DATE : <i>25.02.97</i>	
VIDE DE SALLE VÉRIFIÉ PAR : <i>C5</i>	DATE : <i>26.02.97</i>	
STATUT	CONFORME <input checked="" type="checkbox"/>	NON CONFORME <input type="checkbox"/>
NETTOYAGE EFFECTUÉ PAR : <i>C5</i>	DATE : <i>26.02.97</i>	
NETTOYAGE VÉRIFIÉ PAR : <i>RC</i>	DATE : <i>26.02.97</i>	
STATUT	CONFORME <input checked="" type="checkbox"/>	NON CONFORME <input type="checkbox"/>

RC



036  
26.02.97

Dissolution LSNa 902M -  
1000 ml - 75 rpm - COMPAIMES

1- Préparation du milieu de dissolution -

- Pesée de l'eau purifiée le 26.02.97 - Balance OALOM

Tare = 2,725 Kg RC 26.02.97 ~~26/02/92~~

Brut = 19,550 Kg RC 26.02.97 ~~26/02/92~~

Brut - Tare = Net = 19,550 - 2,725 = 16,825 Kg RC 26.02.97  
~~26/02/92~~

- Pesée de LSNa au 1768 - Balance GALOM

16,825 x 288,4 x 0,02 = 97,05 g de LSNa -

**PESEE SIMPLE**

Date de la pesée : 1997/02/26 16:08:27



\*ARR1768 \*

PRODUIT	=>	NALAUSEF
POIDS NET	=>	0.097 KG
TARE	=>	0.175 KG
POIDS BRUT	=>	0.272 KG

RC

FOURNIER 1001516







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## 2- Remplissage des ~~flacons~~ bords de dissolution -

037

Balance GAL ~~M~~ Dissolutest GAL 026 - Prélabo.

1 L L8Na 902M pèse 1000g -

bol	mise à zéro	Pesée du L8Na 902M	Signatures
1	oui	1000,0 g	26/02/97  RC 26.02.97
2	oui	1000,0 g	26/02/97  RC 26.02.97
3	oui	1000,0 g	26/02/97  RC 26.02.97
4	oui	1000,0 g	26/02/97  RC 26.02.97
5	oui	1000,0 g	26/02/97  RC 26.02.97
6	oui	1000,0 g	26/02/97  RC 26.02.97

## 3- Pesée des comprimés - Balance AG 204 - GAL 205 -

Code	340-100	1	0.0 mg	2	0.0 mg	3	0.0 mg	4	0.0 mg	5	0.0 mg	6	0.0 mg	RC
26.02.97	17:47:06		465.8 mg		458.9 mg		461.8 mg		456.6 mg		460.0 mg		450.2 mg	
Code				Code		Code		Code		Code		Code		

## 4- Conditions.

Palettes ~~700~~ 71 RC

75 TPM RC 27.02.97 RC 27/2/97

37°C ± 0,5°C RC 27.02.97 RC 27/2/97.

λ = 290 nm - UV GAL 233 - UVI KON ~~233~~ - Cure 2mm -

Prélèvement manuel - 5 ml - remplacement -

Seringue + filtre ref 178 398501 - Filtration sur  
filtre Millex HA ref 8LHA025NB avant lecture -

FOURNIER 1001517

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M-11.07.97

038

LONGUEUR D ONDE FIXE 290 NM CPR LSNA 0.02M 75 RPM

RL

02-27-1997 10:37

Lambda	No.	Valeur_E
290.0	1	0.0000_1 <i>Ain / Ain</i>
290.0	2	-0.0027_1 <i>LSNA / LSNA</i>
290.0	3	-0.0016_1
290.0	4	-0.0025_1
290.0	5	-0.0019_1
290.0	6	-0.0019_1 <i>0 min</i>
290.0	7	-0.0021_1
290.0	8	-0.0016_1
290.0	9	0.3039_1
290.0	10	0.3059_1
290.0	11	0.2762_1
290.0	12	0.3613_1 <i>5 min</i>
290.0	13	0.3231_1
290.0	14	0.4628_1
290.0	15	0.6251_1
290.0	16	0.6282_1
290.0	17	0.6157_1
290.0	18	0.6211_1 <i>10 min</i>
290.0	19	0.6286_1
290.0	20	0.6641_1
290.0	21	0.7101_1
290.0	22	0.7102_1
290.0	23	0.7219_1
290.0	24	0.7096_1 <i>15 min</i>
290.0	25	0.7073_1
290.0	26	0.7254_1
290.0	27	0.7708_1
290.0	28	0.7608_1
290.0	29	0.7666_1
290.0	30	0.7483_1 <i>20 min</i>
290.0	31	0.7512_1
290.0	32	0.7504_1
290.0	33	0.8151_1
290.0	34	0.8141_1
290.0	35	0.8261_1 <i>30 min</i>
290.0	36	0.7999_1
290.0	37	0.8109_1
290.0	38	0.7943_1
290.0	39	0.8324_1
290.0	40	0.8430_1
290.0	41	0.8489_1
290.0	42	0.8324_1 <i>40 min</i>
290.0	43	0.8379_1
290.0	44	0.8218_1
290.0	45	0.8621_1
290.0	46	0.8354_1
290.0	47	0.8739_1
290.0	48	0.8458_1 <i>50 min</i>
290.0	49	0.8512_1
290.0	50	0.8365_1
290.0	51	0.8792_1
290.0	52	0.8629_1
290.0	53	0.8850_1 <i>60 min</i>
290.0	54	0.8552_1
290.0	55	0.8672_1
290.0	56	0.8492_1

FOURNIER 1001518

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KONTRON INSTRUMENTS UVIKON 922

Opérateur .....

12 11 2 512

# DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 27/02/97  
APPAREIL : GAL233GAL086  
LONGUEUR D'ONDE : 290nm  
CUVE en mm : 2mm

TITRE : Comprimés PHARMA PASS LOT 340  
N° CAHIER : LF 178TER dissolution n°1 p 036  
FICHIER : m:\commun\glnq\donbase\lf178ter\dissol\340b  
ELUANT : Lsna 0,02M  
AGITATION : 75 RPM

## PREPARATION DES ECHANTILLONS

masse théorique 459  
dosage théorique 100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	465,80	458,90	461,80	456,60	460,00	450,10
quantité de principe actif	101,48	99,98	100,61	99,48	100,22	98,06

Témoin 100mg 0,900

Fournier 1001519

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## SUIVI DE LA DISSOLUTION

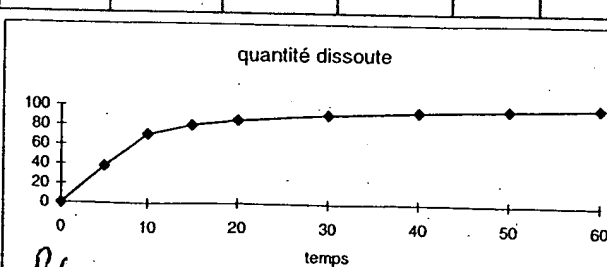
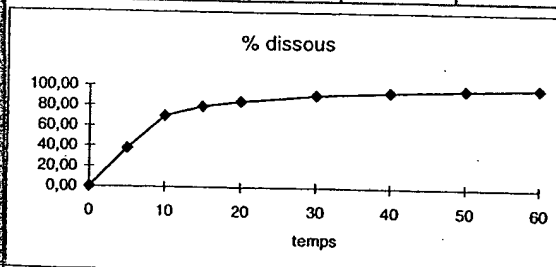
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	1000	0	0	0	0	0	0
5	1000	0,304	0,306	0,276	0,361	0,323	0,463
10	1000	0,625	0,628	0,616	0,621	0,629	0,664
15	1000	0,710	0,710	0,722	0,710	0,707	0,725
20	1000	0,771	0,761	0,767	0,748	0,751	0,750
30	1000	0,815	0,814	0,826	0,800	0,811	0,794
40	1000	0,832	0,843	0,849	0,832	0,838	0,822
50	1000	0,862	0,835	0,874	0,846	0,851	0,837
60	1000	0,879	0,863	0,885	0,855	0,867	0,849

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	37,73	33,28	34,01	30,48	40,32	35,81	52,46	7,92	20,99
10,0	70,29	68,60	69,99	68,18	69,56	69,92	75,50	2,65	3,77
15,0	79,91	78,25	79,43	80,23	79,85	78,91	82,79	1,57	1,97
20,0	85,18	85,31	85,49	85,60	84,49	84,18	86,03	0,70	0,83
30,0	91,38	90,55	91,80	92,54	90,72	91,25	91,44	0,73	0,80
40,0	94,73	92,86	95,48	95,53	94,74	94,69	95,06	0,98	1,04
50,0	96,84	96,60	95,06	98,76	96,77	96,60	97,23	1,20	1,23
60,0	99,03	98,93	98,63	100,46	98,25	98,85	99,06	0,76	0,76
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	37,65	33,78	34,00	30,67	40,11	35,89	51,44	7,44	19,75
10,0	70,25	69,61	69,97	68,60	69,20	70,07	74,04	1,93	2,75
15,0	79,87	79,41	79,41	80,72	79,43	79,08	81,18	0,86	1,07
20,0	85,16	86,58	85,47	86,12	84,05	84,37	84,36	1,05	1,23
30,0	91,36	91,89	91,78	93,10	90,24	91,45	89,67	1,23	1,35
40,0	94,70	94,24	95,46	96,12	94,24	94,90	93,22	1,02	1,08
50,0	96,81	98,03	95,03	99,36	96,26	96,81	95,34	1,65	1,70
60,0	99,00	100,40	98,61	101,07	97,73	99,06	97,14	1,51	1,53
0,0									
0,0									
0,0									
0,0									



M 11.02.97

RL

U411  
26.02.97

Dissolution LSNa 0,02M -  
1000 ml - 75 rpm - GELULES

1. Préparation du milieu de dissolution.  
cf page 036.

FOURNIER 1001520  
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Subject to  
Protective Order

2. Remplissage des bobs de dissolution - Balance GAL 111 -  
Balance GAL 111 - Dissolutest AT7 Solar GAL 091  
1 l LSNa 0,02M pèse 1000,0g

bob	mise à zéro	Pesée de LSNa 0,02M.	Signature.
1	oui	1000,0 g	26/02/97 <i>DR</i> Re 26.02.97
2	oui	1000,0 g	26/02/97 <i>DR</i> Re 26.02.97
3	oui	1000,0 g	26/02/97 <i>DR</i> Re 26.02.97
4	oui	1000,0 g	26/02/97 <i>DR</i> Re 26.02.97
5	oui	1000,0 g	26/02/97 <i>DR</i> Re 26.02.97
6	oui	1000,0 g	26/02/97 <i>DR</i> Re 26.02.97

3. Pesée des gélules - Balance AG 204 GAL 205

26.02.97 Code	17:50:55 0011710 Code	1	0.0 mg	2	0.0 mg	3	0.0 mg	4	0.0 mg	5	0.0 mg	6	0.0 mg	<i>RC</i>
			430.6 mg		425.6 mg		431.8 mg		426.4 mg		425.0 mg		428.9 mg	
			Code		Code		Code		Code		Code			

4. Conditions

Palettes 75 rpm RC 14.02.97 ce 27/2/97  
37°C ± 0,5°C RC 24.02.97 ce 27/2/97  
m 11.02.97

Prélèvement manuel - 5 ml - remplacement seringue +  
filtre ref 178 39 8501 - filtration sur filtre Miller ref  
3LHA025NB avant lecture.

LONGUEUR D ONDE FIXE 290 NM GELULE LIP200 ARR1710 LSNA 0.02M 75 RPM

02-27-1997 12:54

		Lambda	No.	Valeur_E
RC	RC	290.0	1	0.0000_1 Air/Air
		290.0	2	-0.0047_1 LSNA/LSNA
		290.0	3	-0.0037_1
		290.0	4	-0.0038_1
		290.0	5	-0.0031_1
		290.0	6	-0.0002_1 0 min
		290.0	7	-0.0037_1
		290.0	8	-0.0030_1
		290.0	9	0.1357_1
		290.0	10	0.1093_1
		290.0	11	0.1094_1 5 min
		290.0	12	0.1485_1
		290.0	13	0.1059_1
		290.0	14	0.0899_1
		290.0	15	0.4770_1
		290.0	16	0.4287_1
		290.0	17	0.4325_1
		290.0	18	0.4869_1 10 min
		290.0	19	0.3954_1
		290.0	20	0.4437_1
RC	RC	290.0	21	0.6538_1 n=6 * erreur lecture (Gau lieu de 1)
		290.0	22	0.6836_1
		290.0	23	0.6558_1
		290.0	24	0.6720_1 15 min
		290.0	25	0.6938_1
		290.0	26	0.6299_1
		290.0	27	0.7886_1
		290.0	28	0.7697_1
		290.0	29	0.7797_1 20 min
		290.0	30	0.7786_1
		290.0	31	0.7503_1
		290.0	32	0.7804_1
		290.0	33	0.9695_1
		290.0	34	0.9400_1
		290.0	35	0.9613_1
		290.0	36	0.9389_1 30 min
		290.0	37	0.9283_1
		290.0	38	0.9573_1
		290.0	39	1.0606_1
		290.0	40	1.0423_1
RC	RC	290.0	41	1.0671_1
		290.0	42	1.0249_1 40 min
		290.0	43	1.0207_1
		290.0	44	1.0515_1
		290.0	45	1.1318_1
		290.0	46	1.1029_1
		290.0	47	1.1333_1
		290.0	48	1.0888_1 50 min
		290.0	49	1.0959_1
		290.0	50	1.0957_1

FOURNIER 1001521  
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Protective Order

M 11.07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
 DATE : 27/02/97  
 APPAREIL : GAL233GAL091  
 LONGUEUR D'ONDE : 290nm  
 CUVE en mm : 2mm

TITRE : Gelule LIP 200 usine arr1710  
 N° CAHIER : LF 178TER dissolution n°1 p 040  
 FICHER : m:\commun\glnq\donnbase\lf178ter\disso\arr1710b  
 ELUANT : Lsna 0,02M  
 AGITATION : 75 RPM

FOURNIER 1001522

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## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

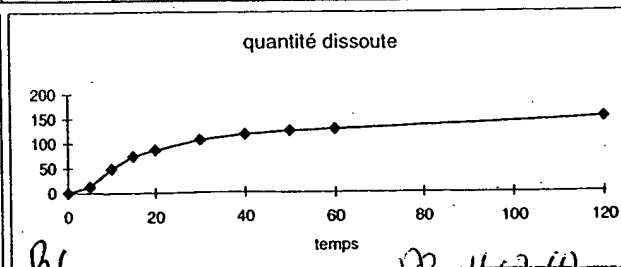
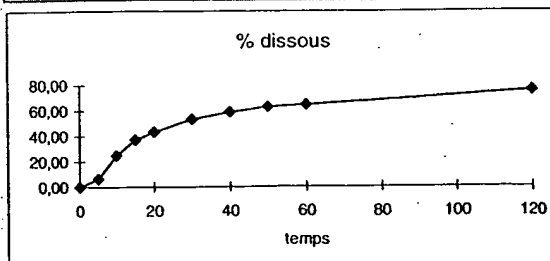
volume prélevé en ml		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	1000	0	0	0	0	0	0
5	1000	0,136	0,109	0,109	0,149	0,106	0,090
10	1000	0,477	0,429	0,433	0,487	0,395	0,444
15	1000	0,684	0,656	0,672	0,694	0,630	0,654
20	1000	0,789	0,770	0,780	0,779	0,750	0,780
30	1000	0,970	0,940	0,961	0,939	0,928	0,957
40	1000	1,061	1,042	1,067	1,025	1,021	1,052
50	1000	1,132	1,103	1,133	1,089	1,096	1,096
60	1000	1,155	1,122	1,147	1,142	1,122	1,115
120	1000	1,325	1,294	1,361	1,335	1,323	1,349

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	6,47	7,56	6,06	6,06	8,28	5,89	5,00	1,21	18,65
10,0	24,71	26,54	23,86	24,09	27,10	21,97	24,69	1,88	7,60
15,0	37,10	38,17	36,59	37,48	38,73	35,14	36,48	1,30	3,50
20,0	43,38	44,19	43,11	43,67	43,65	41,98	43,66	0,77	1,76
30,0	53,29	54,47	52,77	53,94	52,75	52,08	53,71	0,90	1,68
40,0	58,86	59,79	58,70	60,10	57,79	57,50	59,26	1,06	1,79
50,0	62,67	64,03	62,37	64,06	61,63	61,95	61,99	1,09	1,74
60,0	64,41	65,62	63,74	65,15	64,88	63,70	63,35	0,93	1,44
120,0	75,69	75,39	73,60	77,36	75,92	75,18	76,66	1,30	1,72
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	12,94	15,11	12,11	12,11	16,56	11,78	10,00	2,41	18,65
10,0	49,42	53,08	47,73	48,17	54,19	43,95	49,38	3,76	7,60
15,0	74,20	76,34	73,19	74,97	77,46	70,28	72,96	2,60	3,50
20,0	86,76	88,39	86,22	87,34	87,29	83,96	87,33	1,53	1,76
30,0	106,57	108,94	105,54	107,89	105,51	104,16	107,43	1,79	1,68
40,0	117,71	119,59	117,39	120,20	115,58	115,01	118,51	2,11	1,79
50,0	125,35	128,07	124,75	128,12	123,26	123,91	123,99	2,18	1,74
60,0	128,82	131,25	127,47	130,31	129,76	127,40	126,71	1,86	1,44
120,0	151,37	150,78	147,21	154,72	151,84	150,36	153,33	2,61	1,72
0,0									
0,0									
0,0									



RL

MD 11-07-97

28.02.97

Dissolution LSNa 0,025 M.  
Comprimés lot 340

757M

U4

1. Matière première

comprimés de pinopibrate 100mg

lot # 340 date 02/07/97 Pherma Pass

2. Préparation du milieu de dissolution

eau purifiée du jour :

tare = 3,3922 kg. CC 28/02/97 M 28.02.97

brut = 11,1931 kg. CC 28/02/97 M 28.02.97

net = brut - tare = 11,1931 - 3,3922 = 7,8069 kg.

LSNa au 1768 :

FICHE DE NETTOYAGE

LOCAL N° : Salle balances

DATE DE DEBUT D'UTILISATION DU LOCAL : 27.02.97

PRODUIT : LF 178TER

N° DE LOT : 2391/00RC

OPERATION : pesées

DATE DE FIN D'OPERATION : 27.02.97

VIDE DE SALLE EFFECTUÉ PAR : RC

VIDE DE SALLE VÉRIFIÉ PAR : CS

STATUT CONFORME / X

NON CONFORME /   

NETTOYAGE EFFECTUÉ PAR : CS

DATE : 27.02.97

DATE : 28.02.97

NETTOYAGE VÉRIFIÉ PAR : CC

DATE : 28.02.97

DATE : 28/02/97

STATUT CONFORME / X

NON CONFORME /   

97



28.02.97

043

Dissolution LSNa 0,025 M  
Comprimés lot 340

75TPN

1. Matière première

comprimés de finopibrate 100mg

lot # 340 date 02/07/97 Pharmed Pass

2. Préparation du milieu de dissolution

eau purifiée du jour :

tare = 3,3922 kg . CC 88/02/97 M 28.02.97

brut = 11,1931 kg CC 28/02/97 M 28.02.97

net = brut - tare = 11,1931 - 3,3922 = 7,8069 kg .

LSNa au 1768 :

$7,8069 \times 288,4 \times 0,025 = 56,8 \text{ g à peser}$

**PESEE SIMPLE**

Date de la pesée : 1997/02/28 10:26:34



\*ARR1768 \*

PRODUIT	=>	NALAUSEF
POIDS NET	=>	0.056 KG
TARE	=>	0.170 KG
POIDS BRUT	=>	0.226 KG

FOURNIER 1001524  
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Subject to  
Protective Order

RC 03.07.97

### 3. Pesée des bols de dissolution

1 l de LSNa 0,025 N soit une pesée de 1001,0 g

#### 4. Pesée des comprimés

FOURNIER 1001525

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Protective Order**

### 5. Conditions de dissolution

. dissolutest Pro Labo 6AL 103

• UV 930 GAL 108

DE 27/12/97 } palettas 75TPN  
CC 28/01/97 } bain-marie 37 °C ± 0,5 °C

$\lambda = 290 \text{ nm}$   
Caves de  $2 \text{ mm}$ .

• prélèvement 5ml et remplacement par du milieu neuf  
prélèvement avec seringue + filtre Prodox ref 17839 8501

• dosage après filtration sur filtre Millex HA 0,45  $\mu$ m  
ref SLHA 025 NB.

81.03.02.92

6. Mesure

041

COMPRIMES DE FENDOFIBRATE 100 MG PHARMA PASS LOT 340 DANS LSNA 0,025M

28-02-1996 15:2

Lambda	No.	Valeur_E	
290.0	1	-0.0019_1	
290.0	2	-0.0033_1	
290.0	3	0.0000_1	T0
290.0	4	-0.0011_1	
290.0	5	-0.0013_1	
290.0	6	-0.0012_1	
290.0	7	0.1686_1	
290.0	8	0.2075_1	
290.0	9	0.3357_1	T5
290.0	10	0.3257_1	
290.0	11	0.2954_1	
290.0	12	0.4166_1	
290.0	13	0.6367_1	
290.0	14	0.6498_1	
290.0	15	0.6619_1	T10
290.0	16	0.6461_1	
290.0	17	0.6525_1	
290.0	18	0.7147_1	
290.0	19	0.7633_1	
290.0	20	0.7605_1	
290.0	21	0.7598_1	T15
290.0	22	0.7626_1	
290.0	23	0.7713_1	
290.0	24	0.7903_1	
290.0	25	0.8144_1	
290.0	26	0.8045_1	
290.0	27	0.8093_1	
290.0	28	0.8102_1	T20
290.0	29	0.8163_1	
290.0	30	0.8132_1	
290.0	31	0.8591_1	
290.0	32	0.8514_1	
290.0	33	0.8415_1	
290.0	34	0.8717_1	T30
290.0	35	0.8498_1	
290.0	36	0.8542_1	
290.0	37	0.8898_1	
290.0	38	0.8661_1	
290.0	39	0.8662_1	
290.0	40	0.8870_1	T40
290.0	41	0.8915_1	
290.0	42	0.8848_1	
290.0	43	0.9028_1	
290.0	44	0.8867_1	
290.0	45	0.8714_1	
290.0	46	0.8952_1	T50
290.0	47	0.9002_1	
290.0	48	0.8885_1	
290.0	49	0.9064_1	
290.0	50	0.8843_1	
290.0	51	0.8795_1	
290.0	52	0.8942_1	T60
290.0	53	0.8996_1	
290.0	54	0.8822_1	

FOURNIER 1001526

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# DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : C.COGET  
DATE : 28/02/97  
APPAREIL : gal108-gal103  
LONGUEUR D'ONDE : 290nm  
CUVE en mm : 2mm

TITRE : comprimés Fenofibrate 100mg Pharma Pass lot 340  
N° CAHIER : LF178 TER n°1  
FICHIER : m:\commun\glnq\donnbase\LF178TER\DISSOLUTION\340c  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM,

## PREPARATION DES ECHANTILLONS

masse théorique	100
dosage théorique	100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	100,00	100,00	100,00	100,00	100,00	100,00
quantité de principe actif	100,00	100,00	100,00	100,00	100,00	100,00

Témoin 100mg/l 0,900

FOURNIER 1001527  
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## SUIVI DE LA DISSOLUTION

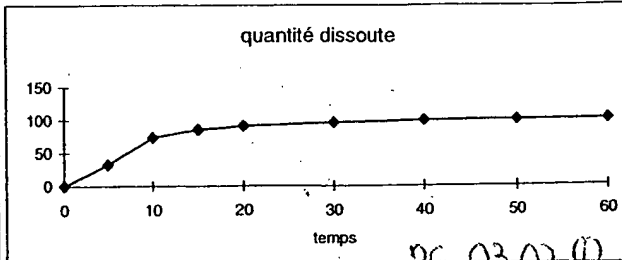
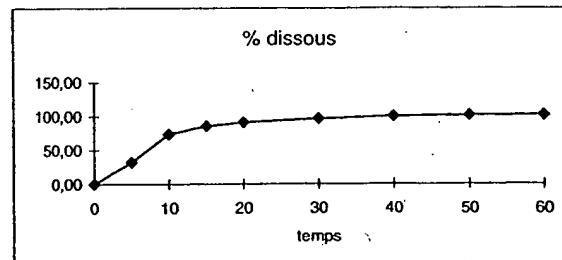
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,169	0,208	0,336	0,326	0,295	0,417
10	1000	0,637	0,650	0,662	0,646	0,653	0,715
15	1000	0,763	0,761	0,760	0,763	0,771	0,790
20	1000	0,814	0,805	0,809	0,810	0,816	0,813
30	1000	0,859	0,851	0,842	0,872	0,850	0,854
40	1000	0,890	0,866	0,866	0,887	0,892	0,885
50	1000	0,903	0,887	0,871	0,895	0,900	0,889
60	1000	0,906	0,884	0,880	0,894	0,900	0,882

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	32,43	18,78	23,11	37,33	36,22	32,78	46,33	10,05	31,00
10,0	73,55	70,87	72,34	73,74	71,96	72,72	79,68	3,14	4,27
15,0	85,86	85,23	85,03	85,00	85,32	86,19	88,41	1,32	1,54
20,0	91,09	91,32	90,34	90,87	90,96	91,62	91,40	0,46	0,50
30,0	96,37	96,77	95,90	94,98	98,30	95,85	96,41	1,12	1,17
40,0	99,77	100,69	98,04	98,12	100,45	100,99	100,33	1,33	1,33
50,0	101,35	102,63	100,86	99,15	101,84	102,38	101,26	1,27	1,25
60,0	101,87	103,46	101,02	100,64	102,22	102,88	100,98	1,16	1,14
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	32,43	18,78	23,11	37,33	36,22	32,78	46,33	10,05	31,00
10,0	73,55	70,87	72,34	73,74	71,96	72,72	79,68	3,14	4,27
15,0	85,86	85,23	85,03	85,00	85,32	86,19	88,41	1,32	1,54
20,0	91,09	91,32	90,34	90,87	90,96	91,62	91,40	0,46	0,50
30,0	96,37	96,77	95,90	94,98	98,30	95,85	96,41	1,12	1,17
40,0	99,77	100,69	98,04	98,12	100,45	100,99	100,33	1,33	1,33
50,0	101,35	102,63	100,86	99,15	101,84	102,38	101,26	1,27	1,25
60,0	101,87	103,46	101,02	100,64	102,22	102,88	100,98	1,16	1,14
0,0									
0,0									
0,0									
0,0									



RC 03.07.97

Re 4/03/97. U4

Demolition LSNa 0,025M.  
gélules Epantyl 200. Au 17/10

1. Préparation du milieu de dissolution LSNa 0,025M.

masse nominale du LSNa = 288,4 g.

pesée de l'eau : tare : 2,755 kg Re 4/3/97. CC 4/03/97

net : 15,170 kg Re 4/3/97. CC 4/03/97

net : 15,170 - 2,755 = 12,415 kg.

soit  $12,415 \times 0,025 \times 288,4 = 89,5$  g de LSNa à peser.

**PESEE SIMPLE**

Date de la pesée : 1997/03/04 10:30:29



PRODUIT => NALAUSF  
POIDS NET => 0.090 KG  
TARE => 0.264 KG  
POIDS BRUT => 0.354 KG

FOURNIER 1001528

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Subject to  
Protective Order

2. Pesée du milieu de dissolution gélules.

1 litre de LSNa 0,025M = 1001,0 g.

bol	gél	Pesée	Signature
1	oui	1001,0g	RC 04.03.97 Re
2	oui	1001,0g	RC 04.03.97 Re
3	oui	1001,0g	RC 04.03.97 Re
4	oui	1001,0g	RC 04.03.97 Re
5	oui	1001,0g	RC 04.03.97 Re
6	oui	1001,0g	RC 04.03.97 Re RC 03.07.97

3. Resaca das gâchelas gâchelos.

011710  
13:24:55

Code 04.03.97

1	ID	0.0000 g
2	ID	0.4315 g
3	ID	0.0000 g
4	ID	0.4296 g
5	ID	0.0000 g
6	ID	0.4278 g
		0.0000 g
		0.4296 g

#### 4. Conditions de dissolution

denoluxer PROUBO gal 103.

$$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$$

AG 4/3/97

CC 4/03/97

8. 75 WPM

BB-413137

CC 4103/97

prélevement de S11 avec remplacement du nœud prélevé  
par S11 de nœud neuf. Utilisation d'une saignée  
plastique de 10 l ref Plastipack 32188 munie d'un  
pistolet ref ProLab 178338501.

filtration awal menggunakan MILLEX HA 0.45  $\mu$ m di Millipore  
SLHA025NB.

S. Mexico.

spectrophotomètre monochromateur 520 gpl. 103 avec cuves de 2 mm.  
de trajet optique.

**FOURNIER 1001529**

**Highly Confidential  
Subject to  
Protective Order**

RC 03.07.97

Liste Paramètre

04-03-1996 13:2

LONGUEUR D'ONDE[nm]..... 290.0  
 TEMPS D'ATTENTE [s]..... 0.0  
 TEMPS INTEGRATION [s]..... 5.0  
 NOMBRE D'ECHANTILLONS..... 1

MODE CALC..... Non

CHANGEMENT LAMPES[nm]..... 340  
 LAMPE DEUTERIUM..... Oui  
 LAMPE TUNGSTENE..... Oui  
 FENTE(S) [nm]..... 2.0  
 IMPRESSION AUTO..... Non  
 SAUVEGARDE AUTO..... Non

KONTRON INSTRUMENTS

UVIKON 93

LIPANTHYL 200M GELULES ARR1710 75TPM LSNA 0.025M

04-03-1996 15:52

Lambda	No.	Valeur_E			
290.0	1	-0.0001_1	A2 au / au'		
290.0	2	-0.0039_1	LS Na / LS Na		
290.0	3	-0.0035_1	A2 LS Na / LS Na		
290.0	4	0.0001_1		290.0	34 1.1278_1
290.0	5	0.0002_1		290.0	35 1.0804_1
290.0	6	0.0001_1		290.0	36 1.1125_1
290.0	7	0.0002_1		290.0	37 1.1338_1
290.0	8	-0.0034_1		290.0	38 1.1376_1
290.0	9	0.0000_1		290.0	39 1.1324_1
290.0	10	0.0730_1		290.0	40 1.2265_1
290.0	11	0.0967_1		290.0	41 1.2169_1
290.0	12	0.1193_1		290.0	42 1.2108_1
290.0	13	0.1561_1		290.0	43 1.1982_1
290.0	14	0.1505_1		290.0	44 1.2338_1
290.0	15	0.1716_1		290.0	45 1.2401_1
290.0	16	0.5148_1		290.0	46 1.2997_1
290.0	17	0.5096_1		290.0	47 1.2857_1
290.0	18	0.5444_1		290.0	48 1.2929_1
290.0	19	0.5483_1		290.0	49 1.3161_1
290.0	20	0.5932_1		290.0	50 1.3020_1
290.0	21	0.5471_1		290.0	51 1.3017_1
290.0	22	0.7831_1		290.0	52 1.3477_1
290.0	23	0.7689_1		290.0	53 1.3109_1
290.0	24	0.7784_1		290.0	54 1.3318_1
290.0	25	0.8115_1		290.0	55 1.3300_1
290.0	26	0.7981_1		290.0	56 1.3411_1
290.0	27	0.8031_1		290.0	57 1.3578_1
290.0	28	0.9336_1		290.0	58 1.4728_1
290.0	29	0.9030_1		290.0	59 1.4518_1
290.0	30	0.9075_1		290.0	60 1.4597_1
290.0	31	0.9370_1		290.0	61 1.4712_1
290.0	32	0.9554_1		290.0	62 1.4719_1
290.0	33	0.9512_1		290.0	63 1.4641_1

FOURNIER 1001530  
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 Protective Order

Re 03 07 97

000

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
 DATE : 04/03/97  
 APPAREIL : gal 103-108  
 LONGUEUR D'ONDE : 290nm  
 CUVE en mm : 2mm

TITRE : Gelule LIP 200 usine arr1710  
 N° CAHIER : LF 178TER dissolution n°1 p 47  
 FICHER : m:\commun\glnq\donnbase\lf178ter\dissolution\arr1710c  
 ELUANT : LSNa 0,025M  
 AGITATION : 75 tpm

## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001531

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## SUIVI DE LA DISSOLUTION

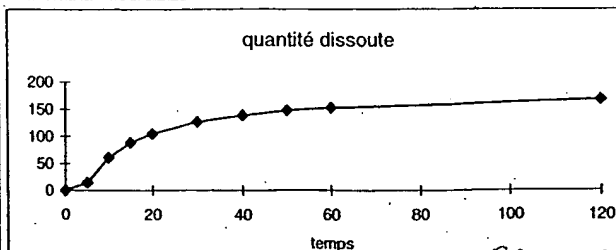
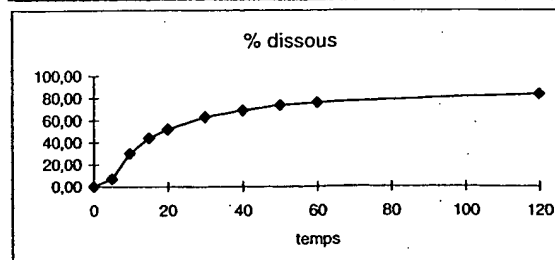
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	1000	0	0	0	0	0	0
5	1000	0,073	0,097	0,119	0,156	0,151	0,172
10	1000	0,515	0,510	0,544	0,548	0,593	0,547
15	1000	0,783	0,769	0,778	0,812	0,798	0,803
20	1000	0,934	0,903	0,908	0,937	0,955	0,951
30	1000	1,128	1,080	1,113	1,134	1,138	1,132
40	1000	1,227	1,217	1,211	1,198	1,234	1,240
50	1000	1,300	1,286	1,293	1,316	1,302	1,302
60	1000	1,348	1,311	1,332	1,330	1,341	1,358
120	1000	1,473	1,452	1,460	1,471	1,472	1,464

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	7,11	4,06	5,39	6,61	8,67	8,39	9,56	2,13	29,88
10,0	30,19	28,63	28,36	30,26	30,49	32,99	30,44	1,66	5,49
15,0	44,10	43,66	42,89	43,41	45,31	44,54	44,81	0,93	2,10
20,0	52,15	52,27	50,55	50,84	52,48	53,48	53,26	1,22	2,33
30,0	62,93	63,31	60,63	62,49	63,68	63,92	63,58	1,23	1,96
40,0	68,82	69,12	68,54	68,24	67,55	69,57	69,89	0,87	1,27
50,0	73,53	73,52	72,72	73,13	74,44	73,69	73,68	0,58	0,79
60,0	75,94	76,54	74,46	75,66	75,58	76,21	77,15	0,93	1,22
120,0	83,45	83,86	82,66	83,14	83,79	83,86	83,42	0,49	0,58
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	14,22	8,11	10,78	13,22	17,33	16,78	19,11	4,25	29,88
10,0	60,39	57,26	56,72	60,51	60,98	65,97	60,87	3,32	5,49
15,0	88,21	87,33	85,78	86,81	90,61	89,08	89,62	1,85	2,10
20,0	104,29	104,54	101,10	101,69	104,95	106,97	106,51	2,43	2,33
30,0	125,87	126,61	121,27	124,97	127,36	127,83	127,15	2,46	1,96
40,0	137,64	138,24	137,09	136,48	135,10	139,13	139,78	1,75	1,27
50,0	147,06	147,03	145,43	146,26	148,88	147,37	147,36	1,17	0,79
60,0	151,87	153,09	148,92	151,31	151,17	152,43	154,30	1,86	1,22
120,0	166,91	167,73	165,32	166,28	167,57	167,73	166,84	0,97	0,58
0,0									
0,0									
0,0									



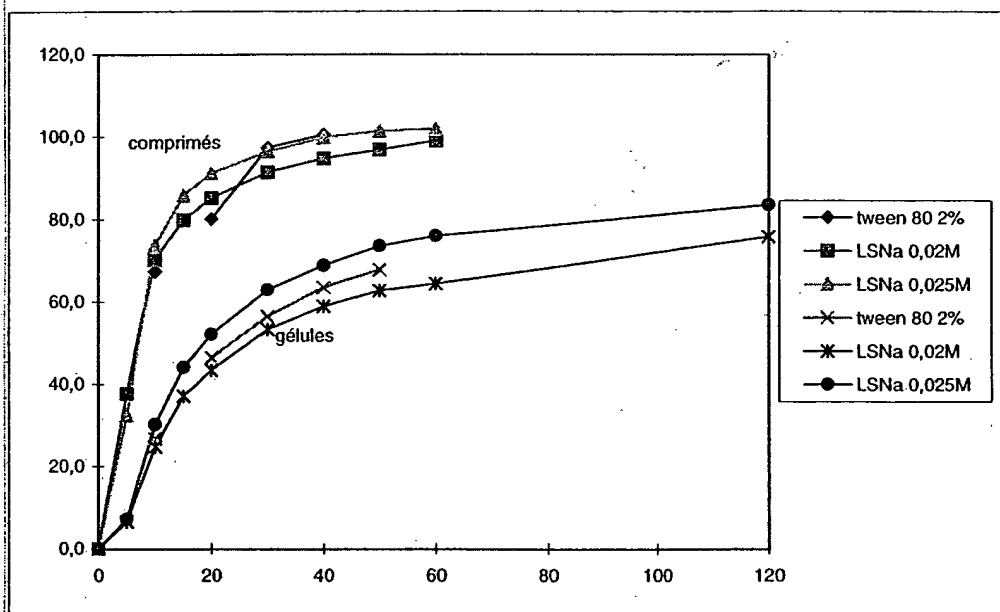
R(11307) 97



**COMPARAISON DES DISSOLUTIONS DU COMPRIME PHARMA PASS  
lot 340 ET DE LA GELULE LIPANTHYL 200M ARR 1710**

temps	comprimés			gélules		
	tween 80 2%	LSNa 0,02M	LSNa 0,025M	tween 80 2%	LSNa 0,02M	LSNa 0,025M
0	0,0	0,0	0,0	0,0	0,0	0,0
5		37,7	32,4		6,5	7,1
10	67,4	70,3	73,6	26,8	24,7	30,2
15		79,9	85,9		37,1	44,1
20	80,1	85,2	91,1	46,4	43,4	52,2
30	97,3	91,4	96,4	56,5	53,3	62,9
40	100,5	94,7	99,8	63,5	58,9	68,8
50		96,8	101,4	67,8	62,7	73,5
60		99,0	101,9		64,4	75,9
120					75,7	83,5

données de base cahier LF178ter p27 à p50



AC 03.07.97

**FOURNIER 1001532**  
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Le 6/3/97

Recherche de la concentration de  
saturation du comiconisat  
dans le LSNa 0,25M.

but du test: rechercher la concentration de saturation du comiconisat  
de fenofibrate Au 1709 dans le LSNa 0,25M.

Protocole: Préparer deux solutions de comiconisat en excès dans  
du LSNa 0,25M. dans fiole jaugée de 1 litre -  
Mettre sous agitation pendant 24h. 24h  
filtrer sur miller 0,45  $\mu$ m ref XH4025W3.  
faire un balayage spectral dans cuve de 2 mm de trajet  
optique.  
appel pour 100 mg/l de feno absorbance = 0,900.

lecture: sur spectrophotomètre KONTRON 922 gal 233.  
LSNa 0,25M dans la cuve de référence -  
selon méthode suivante.

CHANGEMENT LAMPES [nm].....	340
LAMPE DEUTERIUM.....	Oui
LAMPE TUNGSTENE.....	Oui
FENTE(S) [nm].....	2.0
AUTOZERO AU DEPART.....	Non
IMPRESSION AUTO.....	Non
SAUVEGARDE AUTO.....	Non
TRANSFERT AUTO.....	Non

FOURNIER 1001533

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Le 6/3/97

Recherche de la concentration de  
saturation du comiconisat  
dans la LSNa 0,025M.

but du test: rechercher la concentration de saturation du comiconisat  
de feroflinate Au 1709 dans la LSNa 0,025M.

Protocole: Préparer deux solutions de comiconisat en excès dans  
de la LSNa 0,025M dans fiole jaugée de 1 litre.  
Mettre sous agitation pendant 24h.  
Filtrer sur filtres 0,45 µm.  
Faire un balayage spectral dans cuve de 2 mm de trajet  
optique.  
appel pour 100 mg/l de fers absorbance = 0,900.

SOLUTION DE COMICRONISAT DANS LSNa 0.025M SOLUTION B

Liste Paramètre

03-07-1997 09:37

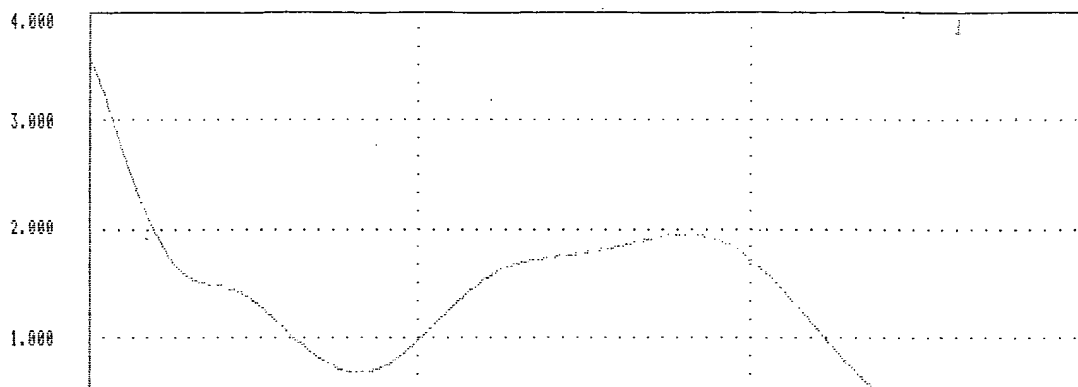
GAMME SPECTRE[nm].....	200-350
VITESSE [nm/min].....	200
INTERVAL MESURE [nm].....	0.5
Nb. DE CYCLES.....	1
TEMPS DE CYCLE [min].....	0.0
NOMBRE D'ECHANTILLONS.....	1
MODE CALC.....	Détection pics
Mode pic.....	Pics
Sensibilité.....	0.1000 ABS
CHANGEMENT LAMPES[nm].....	340
LAMPE DEUTERIUM.....	Oui
LAMPE TUNGSTENE.....	Oui
FENTE(S) [nm].....	2.0
AUTOZERO AU DEPART.....	Non
IMPRESSION AUTO.....	Non
SAUVEGARDE AUTO.....	Non
TRANSFERT AUTO.....	Non

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Ymin = 0.0529 Ymax = 3.5780 file =

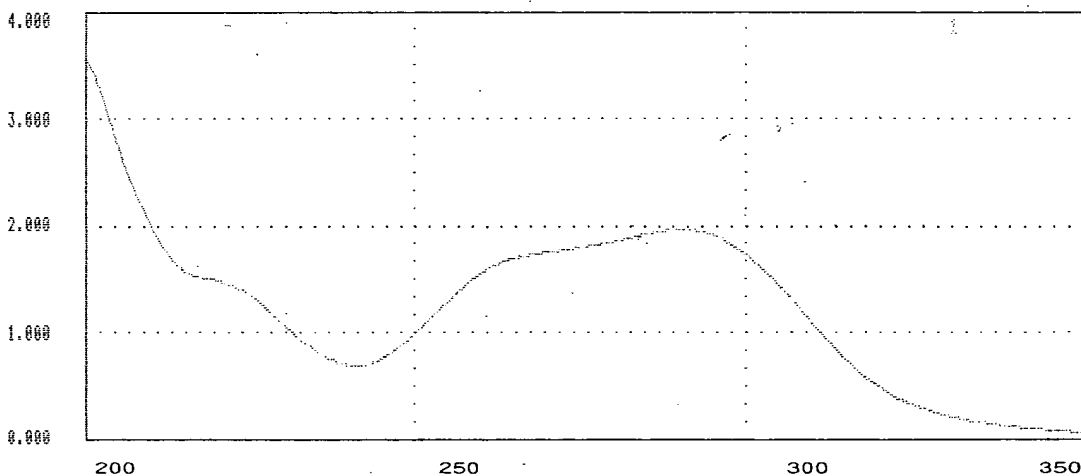
03-07-1997 09:32



## SOLUTION DE COMICRONISAT DANS LSNA0.025M SOLUTION B

Ymin = 0.0565 Ymax = 3.5666 file =

03-07-1997 09:36



## Résultats détection pics

Sensibilité..... 0.1000

Mode sensibilité..... ABS

Ech. 1

Loc  
ValeurPic  
290.50  
1.9681

FOURNIER 1001535

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KONTRON INSTRUMENTS UVIKON 922

Opérateur .....

Solution A :  $CS = \frac{1,954 \times 100}{0,900} = 217,1 \text{ mg de fénofibrate soit}$   
 $\frac{217,1 \times 207}{200} = 224,7 \text{ mg de comiconisat}$

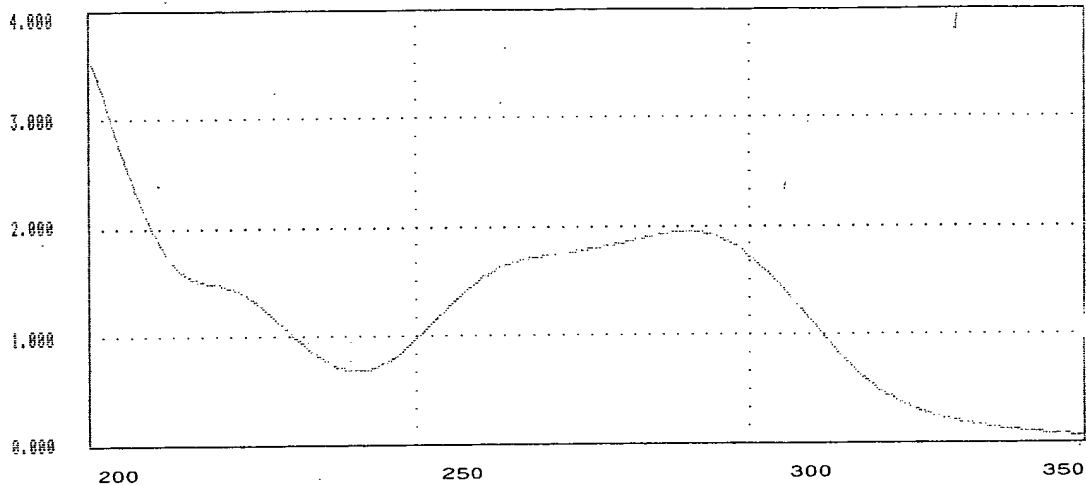
Solution B  $\Rightarrow CS = \frac{1,968 \times 100}{0,900} = 218,7 \text{ mg de fénofibrate soit}$   
 $\frac{218,7 \times 207}{200} = 226,3 \text{ mg de comiconisat}$   
 03.07.97 RC

SOLUTION DE COMICRONISAT DANS LSNA0.025M SOLUTION A

053

03-07-1997 09:32

Ymin = 0.0529 Ymax = 3.5780 file =



Résultats détection pics

Sensibilité..... 0.1000  
Mode sensibilité..... ABS

Ech. 1

Loc  
Valeur

Pic  
290.50  
1.9540

KONTRON INSTRUMENTS UVIKON 922

Opérateur .....

FOURNIER 1001536

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Subject to  
Protective Order

KON

sat.

*guy nade sat*

0192-27

Résultat.

La concentration de saturation du cobicistat Au J709 dans  
le plasma est de  $\left( \frac{224,7 + 226,3}{2} = 225,5 \text{ mg} \right)$

$225,5 \text{ mg/l.}$

celle du fénofibrate est de  $\frac{217,1 + 218,7}{2} = 217,9 \text{ mg/l.}$

03.07.97RC.

FOURNIER 1001537  
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# Dissolution Compimé

Pharma Pass Lot 351.

## 1. Préparation du LSA 0,025H.

Massa totale du LSA = 293,4 g.

pesée de l'eau:

tare 2,785 kg Ae 10/3/97. M 10/03/97

bout 23,550 kg Ae 10/3/97. M 10/03/97

net = 23,550 - 2,785 = 20,765 kg

soit  $20,765 \times 0,025 \times 293,4 = 149,7$  g de LSA à peser.

voir, p deviance pour étiquette

## 2. Pesée du milieu de dissolution gelée.

1 L de LSA 0,025H = 1001,0 g.

lots	Remise à jour	Pesée	Signature.
1	oui	1001,0g	10/03/97 CC Ae 10/3/97
2	oui	1001,0g	10/03/97 CC Ae 10/3/97
3	oui	1001,0g	10/03/97 CC Ae 10/3/97
4	oui	1001,0g	10/03/97 CC Ae 10/3/97
5	oui	1001,0g	10/03/97 CC Ae 10/3/97
6	oui	1001,0g	10/03/97 CC Ae 10/3/97

FOURNIER 1001538

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## 3. Conditions de dissolution.

dissolvent palettes gelées

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$

$\gamma = 75$  rpm

Ae 10/3/97 M 10.03.97

Rélevement de Sol avec remplacement du milieu prélevé par du neuf

Utilisation d'une seringue ref Plastipack 302188 munie d'un  
filtre Probiso ref: 178398501. 02.07.97 AC

PESEE SIMPLE

Date de la pesée : 1997/03/10 09:32:47



\*ARR1768 \*

PRODUIT => NALAUSF  
POIDS NET => 0.150 KG  
TARE => 0.266 KG  
POIDS BRUT => 0.416 KG

4. Pesée des comprimés

balance AB204 g/e

1 comprimé de 434 mg contient 100 mg de fénofibrate.

Code 351

ID		ID		ID	
0.0000 g		0.0000 g		0.0000 g	
0.4336 g		0.4344 g		0.4415 g	
10.03.97	13:06:59				
ID	2	ID	4	ID	6
0.0000 g		0.0000 g		0.0000 g	
0.4395 g		0.4404 g		0.4349 g	

5. lecture

sur spectrophotomètre KONTRON 920 AC03.07.97  
g/e

filtration des échantillons sur ~~filtre~~ filtre Miller séphall pore  
SLHA02SUB.

FOURNIER 1001539

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Protective Order

AC03.07.97



# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 10/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290nm  
CUVE en mm : 2mm

TITRE : comprimé ténofibrate 100mg PHARMA PASS lot 351  
N° CAHIER : LF 178ter p 55  
FICHIER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 351  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

FOURNIER 1001540

## PREPARATION DES ECHANTILLONS

masse théorique 434  
dosage théorique 100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	433,60	439,50	434,40	440,40	441,50	434,90
quantité de principe actif	99,91	101,27	100,09	101,47	101,73	100,21

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

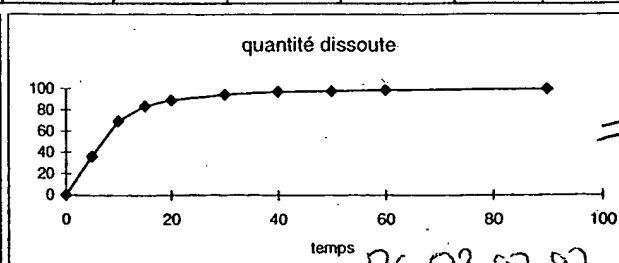
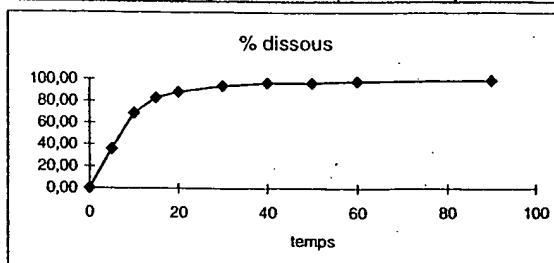
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,357	0,249	0,350	0,347	0,304	0,356
10	1000	0,658	0,598	0,623	0,634	0,595	0,637
15	1000	0,768	0,719	0,717	0,765	0,752	0,762
20	1000	0,807	0,784	0,752	0,814	0,808	0,803
30	1000	0,844	0,814	0,830	0,854	0,845	0,844
40	1000	0,857	0,865	0,847	0,871	0,861	0,856
50	1000	0,863	0,867	0,847	0,874	0,847	0,841
60	1000	0,864	0,869	0,852	0,874	0,870	0,864
90	1000	0,867	0,870	0,852	0,876	0,882	0,859

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	36,09	39,70	27,32	38,85	38,00	33,20	39,47	4,92	13,63
10,0	69,01	73,38	65,75	69,35	69,61	65,15	70,83	3,11	4,51
15,0	82,91	85,98	79,35	80,13	84,30	82,63	85,04	2,70	3,25
20,0	88,55	90,74	86,88	84,42	90,09	89,15	90,01	2,43	2,74
30,0	93,82	95,30	90,60	93,49	94,91	93,64	95,00	1,75	1,86
40,0	96,60	97,22	96,64	95,84	97,24	95,85	96,80	0,63	0,65
50,0	96,74	98,36	97,34	96,31	98,05	94,79	95,61	1,41	1,46
60,0	98,21	98,95	98,03	97,34	98,52	97,76	98,63	0,60	0,62
90,0	98,92	99,77	98,62	97,81	99,22	99,55	98,55	0,73	0,74
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	36,35	39,67	27,67	38,89	38,56	33,78	39,56	4,79	13,17
10,0	69,53	73,31	66,58	69,42	70,64	66,28	70,98	2,72	3,90
15,0	83,55	85,90	80,36	80,21	85,55	84,06	85,22	2,60	3,12
20,0	89,24	90,66	87,98	84,49	91,41	90,70	90,20	2,60	2,92
30,0	94,55	95,22	91,75	93,58	96,31	95,26	95,20	1,63	1,72
40,0	97,35	97,13	97,87	95,93	98,67	97,50	97,00	0,92	0,95
50,0	97,50	98,27	98,57	96,40	99,49	96,43	95,81	1,48	1,52
60,0	98,97	98,86	99,28	97,43	99,98	99,45	98,83	0,87	0,88
90,0	99,69	99,68	99,87	97,90	100,69	101,27	98,76	1,23	1,24
0,0									
0,0									
0,0									



RC 03.07.97

LIP 100 COMPRIME LOT 351

10-03-1996 15:00

Lambda	No.	Valeur_E			
290.0	1	0.0000_1	A2 air / air		
290.0	2	0.0009_1	LSH / LSH		
290.0	3	0.0005_1	A2 LSH / LSH		
290.0	4	-0.0017_1		290.0	34 0.8438_1
290.0	5	-0.0036_1		290.0	35 0.8141_1
290.0	6	-0.0035_1		290.0	36 0.8300_1
290.0	7	-0.0046_1		290.0	37 0.8537_1
290.0	8	-0.0034_1		290.0	38 0.8451_1
290.0	9	0.0000_1		290.0	39 0.8444_1
290.0	10	0.3569_1		290.0	40 0.8565_1
290.0	11	0.2491_1		290.0	41 0.8652_1
290.0	12	0.3498_1		290.0	42 0.8467_1
290.0	13	0.3471_1		290.0	43 0.8705_1
290.0	14	0.3037_1		290.0	44 0.8614_1
290.0	15	0.3555_1		290.0	45 0.8562_1
290.0	16	0.6582_1		290.0	46 0.8630_1
290.0	17	0.5978_1		290.0	47 0.8670_1
290.0	18	0.6225_1		290.0	48 0.8468_1
290.0	19	0.6335_1		290.0	49 0.8740_1
290.0	20	0.5952_1		290.0	50 0.8467_1
290.0	21	0.6365_1		290.0	51 0.8411_1
290.0	22	0.7683_1		290.0	52 0.8637_1
290.0	23	0.7190_1		290.0	53 0.8692_1
290.0	24	0.7167_1		290.0	54 0.8516_1
290.0	25	0.7646_1		290.0	55 0.8735_1
290.0	26	0.7516_1		290.0	56 0.8700_1
290.0	27	0.7622_1		290.0	57 0.8640_1
290.0	28	0.8072_1		290.0	58 0.8674_1
290.0	29	0.7839_1		290.0	59 0.8698_1
290.0	30	0.7517_1		290.0	60 0.8522_1
290.0	31	0.8138_1		290.0	61 0.8757_1
290.0	32	0.8078_1		290.0	62 0.8819_1
290.0	33	0.8025_1		290.0	63 0.8593_1

FOURNIER 1001541

Highly Confidential  
Subject to  
Protective Order

le 11/3/97

Dissolution Comprimé fenofibrate

100 mg PHARMA PASS lot 354

Ces comprimés de 434 mg contiennent 100 mg de fenofibrate.

1. Préparation du LSNa 0,25M.

voir p 55,

FOURNIER 1001542

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2. Pesée du nitro de dissolution gal eu,

1 l de LSNa 0,25M = 1001,0 gr.

lots	rés	Pesée	Signature.
1	oui	1001,0 g.	sz 11/03/97 AB 11/3/97
2	oui	1001,0 g.	sz 11/03/97 AB 11/3/97
3	oui	1001,0 g.	sz 11/03/97 AB 11/3/97
4	oui	1001,0 g.	sz 11/03/97 AB 11/3/97
5	oui	1001,0 g.	sz 11/03/97 AB 11/3/97
6	oui	1001,0 g.	sz 11/03/97 AB 11/3/97.

3. Pesée des comprimés gal los.

11.03.97

09:18:31

Code

354

ID	1	ID	3	ID	5
0.0000 g		0.0000 g		0.0000 g	
0.4345 g		0.4358 g		0.4341 g	
ID	2	ID	4	ID	6
0.0000 g		0.0000 g		0.0000 g	
0.4365 g		0.4345 g		0.4364 g	

RC 03.07.97

4. Conditions de dissolution

dissolvest Profaso à palettes gal 100.

$$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$$

S: 75 RPM

Ag 11/3/97 ML 11.03.07

Prélèvement de sel de milieu avec remplacement par sel de milieu neuf.

Utilisation d'une seringue plastique de 10 ml réf Plastipack 302188 munie d'un piféria Profaso réf 178398501.

5. Lecture.

sur spectrophotomètre KONTRON 922 gal 108.  
cuve de 2 cm de trajet optique,

COMPRIME PHARMA PASS 100MG FENO LOT 354

			290.0	28	0.8138_1	
			290.0	29	0.8229_1	
			290.0	30	0.8159_1	
			290.0	31	0.8242_1	20'
			290.0	32	0.8167_1	
			290.0	33	0.8208_1	
			290.0	34	0.8476_1	
			290.0	35	0.8569_1	
			290.0	36	0.8472_1	20'
			290.0	37	0.8544_1	
			290.0	38	0.8571_1	
			290.0	39	0.8526_1	
			290.0	40	0.8460_1	
			290.0	41	0.8762_1	
			290.0	42	0.8605_1	40'
			290.0	43	0.8633_1	
			290.0	44	0.8636_1	
			290.0	45	0.8675_1	
			290.0	46	0.8540_1	
			290.0	47	0.8746_1	
			290.0	48	0.8634_1	20'
			290.0	49	0.8592_1	
			290.0	50	0.8580_1	
			290.0	51	0.8640_1	
			290.0	52	0.8551_1	
			290.0	53	0.8618_1	
			290.0	54	0.8679_1	60'
			290.0	55	0.8628_1	
			290.0	56	0.8726_1	
			290.0	57	0.8552_1	
			290.0	58	0.8519_1	
			290.0	59	0.8571_1	
			290.0	60	0.8608_1	30'
			290.0	61	0.8582_1	
			290.0	62	0.8595_1	
			290.0	63	0.8622_1	
Lambda	No.	Valeur_E				
290.0	1	0.0000_1	290.0	1	0.0000_1	
290.0	2	0.0027_1	290.0	2	0.0027_1	
290.0	3	-0.0001_1	290.0	3	-0.0001_1	
290.0	4	-0.0043_1	290.0	4	-0.0043_1	
290.0	5	-0.0044_1	290.0	5	-0.0044_1	
290.0	6	-0.0006_1	290.0	6	-0.0006_1	
290.0	7	-0.0006_1	290.0	7	-0.0006_1	
290.0	8	-0.0000_1	290.0	8	-0.0000_1	
290.0	9	-0.0007_1	290.0	9	-0.0007_1	
290.0	10	0.3012_1	290.0	10	0.3012_1	
290.0	11	0.2887_1	290.0	11	0.2887_1	
290.0	12	0.2642_1	290.0	12	0.2642_1	
290.0	13	0.3013_1	290.0	13	0.3013_1	
290.0	14	0.2728_1	290.0	14	0.2728_1	
290.0	15	0.3201_1	290.0	15	0.3201_1	
290.0	16	0.6523_1	290.0	16	0.6523_1	
290.0	17	0.6564_1	290.0	17	0.6564_1	
290.0	18	0.6034_1	290.0	18	0.6034_1	
290.0	19	0.6607_1	290.0	19	0.6607_1	
290.0	20	0.6216_1	290.0	20	0.6216_1	
290.0	21	0.6596_1	290.0	21	0.6596_1	
290.0	22	0.7651_1	290.0	22	0.7651_1	
290.0	23	0.7770_1	290.0	23	0.7770_1	
290.0	24	0.7602_1	290.0	24	0.7602_1	
290.0	25	0.7713_1	290.0	25	0.7713_1	
290.0	26	0.7671_1	290.0	26	0.7671_1	
290.0	27	0.7801_1	290.0	27	0.7801_1	

11-03-1996 12:41

FOURNIER 1001543

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Protective Order

PC 03.01.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 11/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290nm  
CUVE en mm : 2mm

TITRE : comprimé fénofibrate 100mg PHARMA PASS lot 354  
N° CAHIER : LF 178ter p 58  
FICHIER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 354  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

FOURNIER 1001544

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## PREPARATION DES ECHANTILLONS

masse théorique	434
dosage théorique	100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	434,50	436,50	435,80	434,50	434,10	436,40
quantité de principe actif	100,12	100,58	100,41	100,12	100,02	100,55

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

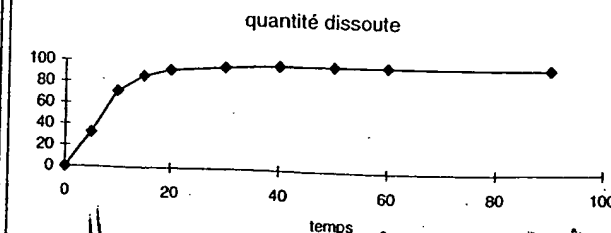
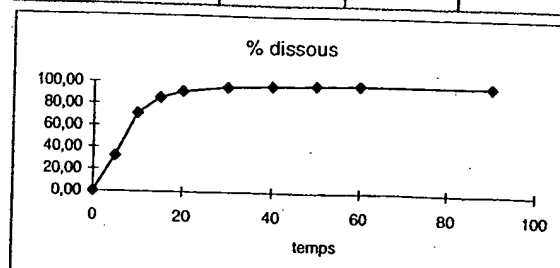
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,301	0,289	0,264	0,301	0	0
10	1000	0,652	0,656	0,603	0,661	0,273	0,320
15	1000	0,765	0,777	0,760	0,771	0,622	0,660
20	1000	0,814	0,823	0,816	0,824	0,767	0,780
30	1000	0,848	0,857	0,847	0,854	0,817	0,821
40	1000	0,846	0,876	0,861	0,863	0,857	0,853
50	1000	0,854	0,875	0,863	0,863	0,864	0,868
60	1000	0,855	0,862	0,868	0,859	0,858	0,864
90	1000	0,852	0,857	0,861	0,858	0,873	0,855

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	32,27	33,41	31,93	29,21	33,41	30,33	35,36	2,25	6,98
10,0	71,32	72,53	72,63	66,87	73,53	69,25	73,11	2,66	3,73
15,0	85,82	85,43	86,36	84,58	86,10	85,70	86,73	0,76	0,89
20,0	91,69	91,29	91,87	91,19	92,41	91,68	91,69	0,44	0,48
30,0	95,86	95,52	96,08	95,07	96,20	96,58	95,68	0,54	0,56
40,0	97,47	95,77	98,66	97,09	97,67	97,83	97,81	0,97	1,00
50,0	97,86	97,12	99,03	97,79	97,71	97,64	97,85	0,63	0,64
60,0	98,39	97,71	98,08	98,82	98,63	99,79	97,33	0,88	0,89
90,0	98,39	97,85	98,00	98,53	98,55	98,83	98,58	0,38	0,38
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	32,37	33,44	32,11	29,33	33,44	30,33	35,56	2,28	7,03
10,0	71,53	72,61	73,05	67,15	73,61	69,26	73,51	2,69	3,76
15,0	86,07	85,53	86,86	84,93	86,20	85,72	87,21	0,86	0,99
20,0	91,97	91,40	92,40	91,57	92,52	91,70	92,20	0,47	0,51
30,0	96,14	95,63	96,64	95,47	96,31	96,60	96,21	0,49	0,51
40,0	97,76	95,88	99,22	97,49	97,78	97,85	98,35	1,11	1,13
50,0	98,15	97,24	99,60	98,20	97,82	97,67	98,39	0,82	0,83
60,0	98,69	97,82	98,64	99,23	98,74	99,81	97,87	0,77	0,78
90,0	98,68	97,96	98,56	98,93	98,66	98,85	99,12	0,40	0,41
0,0									
0,0									
0,0									



RC 03.07.97

# Dissolution comprimé Pharma PASS

100 mg de fexofenadine lot 358

FOURNIER 1001545

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1. Préparation du milieu de dissolution 0,25M  
vau PSS

2. Pesée du milieu de dissolution gal 11.  
1 l de SSC 0,25M = 1001,0g.

lot	gros	Pesée	Signature
1	oui	1001,0g.	ML 11.03.97 de 11/3/97
2	oui	1001,0g	ML 11.03.97 de 11/3/97
3	oui	1001,0g	ML 11.03.97 de 11/3/97
4	oui	1001,0g	ML 11.03.97 de 11/3/97
5	oui	1001,0g.	ML 11.03.97 de 11/3/97
6	oui	1001,0g.	ML 11.03.97 de 11/3/97

3. Pesée des comprimés gal 11

1 comprimé pesé en moyenne 434 mg et contient 100 mg de fexo

11.03.97 14:34:27  
Code 358

1	0.0000 g	2	0.4368 g	3	0.0000 g	4	0.4354 g	5	0.0000 g	6	0.4356 g
ID		ID		ID		ID		ID		ID	

ML 03.07.97

#### 4. Conditions de dissolution

dissolvant Propaso à palettes gel 103.

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$

$\sigma: 75 \text{ rpm}$

> AG 11/3/97 M 11.03.97

Prélèvement pour mesure de S d de riben avec remplacement du riben prélevé par S d de riben neuf.

Utilisation d'une seringue plastique de 20 ml ref Plastipack 302188 munie d'un profilé Propaso ref. 178398501.

#### 5. Lecture

sur spectrophotomètre <sup>930 AC 07.97</sup> ~~roupon 922~~ gel 108, dans cuves de 2 mm de trajet optique.

Filtration des prélèvements sur filtres HA 0,45  $\mu\text{m}$  ref filtres SLHA025 NB.

COMPRIME PHARMA PASS 100MG FEND LOT 358

11-03-1996 16:07

Lambda	No.	Valeur_E
290.0	1	0.0000_1 A2 au/au
290.0	2	-0.0023_1 LSNa/LSNa
290.0	3	0.0002_1 A2 LSNa/LSNa
290.0	4	-0.0004_1
290.0	5	-0.0012_1
290.0	6	-0.0012_1
290.0	7	-0.0013_1 6'
290.0	8	0.0000_1
290.0	9	-0.0000_1
290.0	10	0.3138_1
290.0	11	0.3405_1
290.0	12	0.3417_1
290.0	13	0.4182_1 10'
290.0	14	0.3328_1 5'
290.0	15	0.3469_1
290.0	16	0.6426_1
290.0	17	0.6692_1
290.0	18	0.6698_1
290.0	19	0.6680_1 15'
290.0	20	0.6569_1 10'
290.0	21	0.6768_1

FOURNIER 1001546

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11.03.07.97

290.0 22 0.7360\_1  
290.0 23 0.7620\_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 11/03/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290nm  
CUVE en mm : 2mm

TITRE : comprimé fénofibrate 100mg PHARMA PASS lot 358  
N° CAHIER : LF 178ter p 61  
FICHIER : m:\commun\glnq\donnbase\W178ter\dissolution\lot 358  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

FOURNIER 1001547

Highly Confidential  
Subject to  
Protective Order

### PREPARATION DES ECHANTILLONS

masse théorique	434
dosage théorique	100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	436,80	435,40	435,60	436,50	436,20	438,90
quantité de principe actif	100,65	100,32	100,37	100,58	100,51	101,13

Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

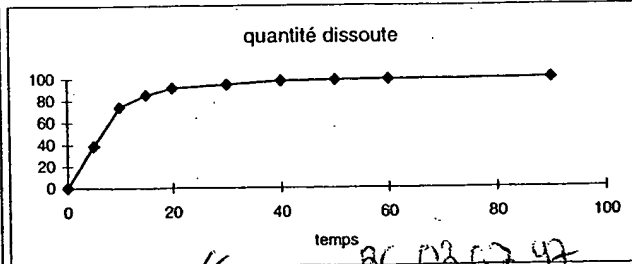
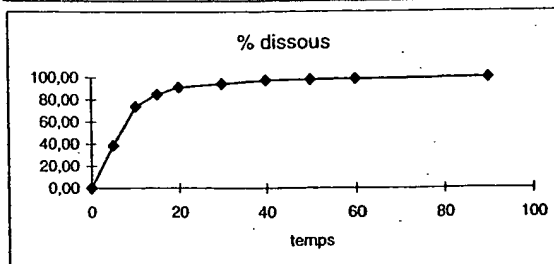
volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
5	1000	0,314	0,341	0,342	0,418	0,333	0,347
10	1000	0,643	0,669	0,670	0,668	0,657	0,677
15	1000	0,756	0,762	0,775	0,785	0,764	0,734
20	1000	0,809	0,816	0,818	0,823	0,808	0,816
30	1000	0,845	0,843	0,808	0,843	0,849	0,846
40	1000	0,859	0,867	0,870	0,866	0,861	0,855
50	1000	0,858	0,867	0,869	0,864	0,865	0,862
60	1000	0,868	0,869	0,861	0,864	0,871	0,867
90	1000	0,869	0,865	0,873	0,867	0,885	0,859

### RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	38,57	34,67	37,77	37,86	46,18	36,81	38,13	3,94	10,21
10,0	73,54	71,16	74,28	74,36	74,03	72,82	74,57	1,32	1,80
15,0	84,81	83,99	84,95	86,35	87,32	85,01	81,21	2,12	2,50
20,0	91,00	90,26	91,36	91,54	91,95	90,29	90,62	0,71	0,78
30,0	94,10	94,68	94,80	90,89	94,62	95,27	94,36	1,60	1,70
40,0	97,22	96,69	97,92	98,20	97,62	97,07	95,82	0,88	0,91
50,0	97,83	97,05	98,40	98,57	97,88	97,99	97,06	0,65	0,66
60,0	98,58	98,63	99,11	98,17	98,36	99,13	98,08	0,46	0,46
90,0	99,39	99,22	99,14	99,97	99,17	101,16	97,68	1,14	1,15
0,0									
0,0									
0,0									

### RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	38,80	34,89	37,89	38,00	46,44	37,00	38,56	3,96	10,21
10,0	73,97	71,62	74,52	74,63	74,45	73,19	75,42	1,36	1,84
15,0	85,30	84,53	85,23	86,67	87,83	85,44	82,12	1,95	2,28
20,0	91,54	90,84	91,65	91,88	92,48	90,75	91,64	0,65	0,71
30,0	94,66	95,29	95,10	91,23	95,16	95,76	95,43	1,70	1,80
40,0	97,79	97,32	98,24	98,56	98,19	97,56	96,90	0,64	0,65
50,0	98,40	97,68	98,72	98,94	98,45	98,48	98,15	0,44	0,45
60,0	99,16	99,27	99,43	98,53	98,93	99,63	99,19	0,39	0,39
90,0	99,98	99,86	99,46	100,34	99,74	101,67	98,78	0,98	0,98
0,0									
0,0									
0,0									





290.0 61 1.3210\_1  
290.0 62 1.3349\_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 07/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2

TITRE : LF 178 TER RG 2394/01 à 18 KG  
N° CAHIER : LF 178 TER n°1 p.98  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2394RG01 18 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	692,80	692,20	694,50	695,70	683,90	685,60
quantité de principe actif	159,63	159,49	160,02	160,30	157,58	157,97

Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

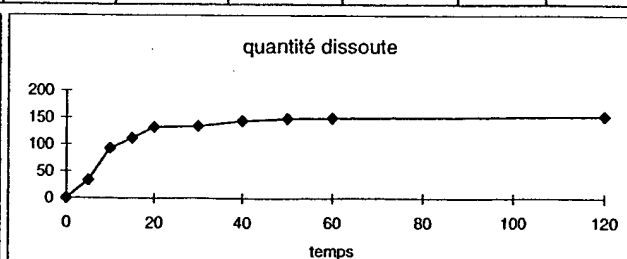
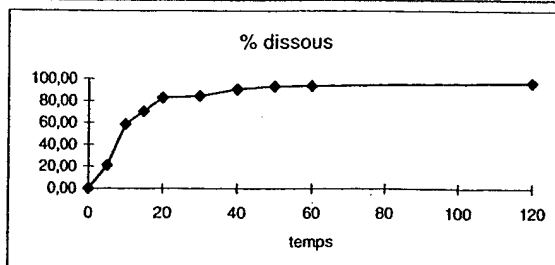
volume prélevé en ml 5							
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,251	0,323	0,295	0,314	0,321	0,308
10	1000	0,774	0,822	0,86	0,863	0,918	0,794
15	1000	1,033	1,02	1,09	0,895	0,897	1,083
20	1000	1,186	1,175	1,174	1,175	1,182	1,174
30	1000	1,267	1,233	1,027	1,246	1,181	1,209
40	1000	1,313	1,286	1,298	1,219	1,276	1,28
50	1000	1,324	1,287	*	1,306	1,304	1,301
60	1000	1,331	1,308	1,309	1,313	1,303	1,277
120	1000	1,366	1,32	*	1,329	1,321	1,335

### RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	21,09	17,47	22,50	20,48	21,76	22,63	21,66	1,93	9,15
10,0	58,65	53,96	57,38	59,82	59,93	64,84	55,95	3,80	6,48
15,0	70,42	72,26	71,46	76,08	62,44	63,68	76,56	6,05	8,60
20,0	82,96	83,27	82,61	82,30	82,16	84,10	83,34	0,74	0,89
30,0	84,50	89,32	87,06	72,50	87,49	84,44	86,22	6,10	7,21
40,0	90,84	92,96	91,18	91,67	86,05	91,56	91,64	2,42	2,67
50,0	93,19	94,18	91,70		92,50	93,98	93,56	1,05	1,13
60,0	93,63	95,13	93,61	92,88	93,44	94,37	92,33	1,01	1,08
120,0	96,18	98,03	94,90		95,01	96,10	96,86	1,31	1,37
0,0									
0,0									
0,0									

### RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	33,56	27,89	35,89	32,78	34,89	35,67	34,22	2,99	8,92
10,0	93,33	86,14	91,51	95,72	96,06	102,18	88,39	5,85	6,26
15,0	112,08	115,35	113,97	121,75	100,10	100,36	120,95	9,67	8,63
20,0	132,04	132,92	131,76	131,69	131,71	132,52	131,66	0,54	0,41
30,0	134,49	142,58	138,86	116,01	140,25	133,07	136,20	9,63	7,16
40,0	144,58	148,40	145,43	146,69	137,94	144,28	144,76	3,58	2,47
50,0	148,16	150,35	146,26		148,28	148,10	147,80	1,46	0,99
60,0	149,03	151,86	149,30	148,64	149,79	148,71	145,86	1,95	1,31
120,0	152,92	156,49	151,36		152,30	151,43	153,01	2,11	1,38
0,0									
0,0									
0,0									



UV GAL 233 - 290 nm - cellule 2mm

LF 178 TER LOT RG 2394/01 18 KG

04-07-1997 02:23

Lambda No. Valeur\_E

1 290.0 1 0.0000\_1  
2 290.0 2 0.0033\_1  
3 290.0 3 0.0001\_1

1 AZ Air / Air

2 LSNr / LSNr

3 AZ LSNr / LSNr

\* valeur aberrante (%)

dehors du T précédent.)

non retenue.

o problème de lecture

nouvelle mesure effectuée

sur le même prélèvement

M 07.06.97.

290.0 T<sub>0</sub> 1 -0.0003\_1  
290.0 2 0.0003\_1  
290.0 3 0.0001\_1  
290.0 4 0.0001\_1  
290.0 5 0.0007\_1  
290.0 6 -0.0002\_1  
290.0 T<sub>5</sub> 7 0.2513\_1  
290.0 8 0.3229\_1  
290.0 9 0.2947\_1  
290.0 10 0.3136\_1  
290.0 11 0.3209\_1  
290.0 12 0.3080\_1  
290.0 T<sub>10</sub> 13 0.7735\_1  
290.0 14 0.8217\_1  
290.0 15 0.8603\_1  
290.0 16 0.8632\_1  
290.0 17 0.9176\_1  
290.0 18 0.7936\_1  
290.0 T<sub>15</sub> 19 1.0326\_1  
290.0 20 1.0200\_1  
290.0 21 1.0899\_1  
290.0 22 0.8948\_1  
290.0 23 0.8965\_1  
290.0 24 1.0830\_1  
290.0 T<sub>20</sub> 25 1.1859\_1  
290.0 26 1.1751\_1  
290.0 27 1.1740\_1  
290.0 28 1.1747\_1  
290.0 29 1.1820\_1  
290.0 30 1.1742\_1

290.0 T<sub>30</sub> 31 1.2671\_1  
290.0 32 1.2328\_1  
290.0 33 1.0270\_1  
290.0 34 1.2457\_1  
290.0 35 1.1814\_1  
290.0 36 1.2088\_1  
290.0 T<sub>40</sub> 37 1.3128\_1  
290.0 38 1.2857\_1  
290.0 39 1.2984\_1  
290.0 40 1.2194\_1  
290.0 41 1.2763\_1  
290.0 42 1.2796\_1  
290.0 T<sub>50</sub> 43 1.3239\_1  
290.0 44 1.2871\_1  
290.0 45 1.2476\_1 - \*  
290.0 46 1.3064\_1  
290.0 47 1.3038\_1  
290.0 48 1.3011\_1  
290.0 T<sub>60</sub> 49 1.3309\_1  
290.0 50 1.3080\_1  
290.0 51 1.3092\_1  
290.0 52 1.2403\_1 - o  
290.0 53 1.0502\_1 - o  
290.0 54 1.3029\_1  
290.0 55 1.2766\_1  
290.0 56 1.3125\_1  
290.0 T<sub>120</sub> 57 1.3662\_1  
290.0 58 1.3197\_1  
290.0 59 1.2825\_1 - \*  
290.0 60 1.3288\_1  
290.0 61 1.3210\_1  
290.0 62 1.3349\_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 07/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2394/01 à 18 KG  
N° CAHIER : LF 178 TER n°1 p.88  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2394RG01 18 kg  
ELUANT : LSNr 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg

masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
692,80 ✓	692,20 ✓	694,50 ✓	695,70 ✓	683,90 ✓	685,60 ✓
159,63	159,49	160,02	160,30	157,58	157,97

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Témoin 100mg/ 0,900

### SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,251 ✓	0,323 ✓	0,295 ✓	0,314 ✓	0,321 ✓	0,308 ✓
10	1000	0,774 ✓	0,822 ✓	0,86 ✓	0,863 ✓	0,918 ✓	0,794 ✓
15	1000	1,033 ✓	1,02 ✓	1,09 ✓	0,895 ✓	0,897 ✓	1,083 ✓
20	1000	1,186 ✓	1,175 ✓	1,174 ✓	1,175 ✓	1,182 ✓	1,174 ✓
30	1000	1,267 ✓	1,233 ✓	1,027 ✓	1,246 ✓	1,181 ✓	1,209 ✓
40	1000	1,313 ✓	1,286 ✓	1,298 ✓	1,219 ✓	1,276 ✓	1,28 ✓
50	1000	1,324 ✓	1,287 ✓				

Re 03.07.97

# Dosage du finofibrate dans

les comprimés du VF178 Ter.

Lot 2394/01 RG - à 14 kg de durée  
- à 18 kg de durée -

Dosage effectué par HPLC selon la procédure 02 DAP004.06/05.

"Identification et dosage du finofibrate dans les gélules à 60 mg de finofibrate micronisé".

## Conditions chromatographiques.

- HPLC gal 087.089.261.
- colonne RP 18 (250 x 4) 5µm. (colonne n°2) Lichrosab réf Merck 50333
- ACN/H<sub>2</sub>O 70/30. réf ACN Merck 41291.  
acidifié à pH=2,5.
- acide orthophosphorique réf ProLabo 2624-295.
- tétrahydrofurane RS réf 28-556-293. ProLabo.
- débit : 1 µl ml/min.
- T° = 35°C
- injection : boucle de 10 µl (la boucle est rincée après injection par H<sub>2</sub>O)
- longueur d'onde : 286 nm.
- temps d'analyse : 10 min pour le STAR.  
11 min pour le CE 5000  
12 min pour le 2020.

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RC03 07 97

## 1. Préparation des solutions témoins gal 25.

### 1.1. solutions témoins mères : (à faire en double)

introduire environ 25,0 mg de finopinate au 124 exactement  
comme dans fiole jaugée de 50 ml.

dissoudre le fino dans 5,0 ml de THF par agitation magnétique.  
qsp avec ACN.

agitation magnétique pdt 30 minutes.

07.04.97

10:41:27

Code

1241

ID

2

ID

1

40540.2 mg raw

soient TH1 et TH2.

36961.6 mg raw

25.6 mg net

25.7 mg net

-40565.9 mg -hwt

-36987.3 mg -hwt //

### 1.2. solutions témoins de travail

à l'aide d'une pipette, introduire 2 ml de solution témoin  
mère dans fiole jaugée de 20 ml.

qsp avec phase mobile puis homogénéiser.

soit T1 à 0,514 µg/10 µl,

T2 à 0,512 µg/10 µl,

## 2. Préparation des solutions essais

### 2.1. solutions essais mères

à faire en double.

broyer soigneusement 10 comprimés dans un mortier. Mélanger.

Peser exactement environ 250,0 mg de mélange dans fiole de 100 ml.

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RC03.07

07.04.97 10:10:52  
ID 2394.01-18.1

07.04.97 10:29:41  
ID 2394.01-14.1

10

85229.2 mg *raie*

85669.8 mg *raie*

351.1 mg *net*

250.1 mg *net*

ID 2394.01-14.2

85579.3 mg *hwt*

-85919.8 mg *hwt*

84795.0 mg *raie*

ID 2394.01-18.2

ID 2394.01-14.1

350.0 mg *net*

82536.9 mg *raie*

85920.2 mg *raie*

-85145.0 mg *hwt*

349.4 mg *net*

101.3 mg *net*

82886.6 mg *hwt*

-86021.6 mg *hwt*

$$2 \times 250,1 + 101,3 = 351,4 \text{ mg}$$

ajouter 15,0 ml de THF et 7,5 ml de phase mobile.

agitation magnétique pour 45 min.

qsp avec ACN.

agitation pour 45 min.

## 2.2. Solutions essais de travail

diluer chaque solution essai de travail mise au 1/25<sup>e</sup>.

qsp avec phase mobile.

## 3. Injections et Résultats.

Les chromatogrammes sont dans l'annexe 2.

\* injecter 5 fois T1 et calculer le facteur de calibration F.

$$F = \frac{CF}{AF}$$

conc en fin de T1 en µg/10 µl

aire du pic.

aire	F.
351611,	$1,462 \times 10^{-6}$
351067,	$1,464 \times 10^{-6}$
351834,	$1,461 \times 10^{-6}$
351263,	$1,463 \times 10^{-6}$
350284,	$1,467 \times 10^{-6}$

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3 RC 03.07.97  
 $\bar{x} = 1,464 \times 10^{-6}$  pour T1

6 RC 03.07.98  
 $\omega = 0,17\%$  satisfaisant

moyenne des 3 derniers essais

$\bar{x} = 1,464 \times 10^{-6}$

RC 03.07.97

calcul du facteur de calibration de T2.

cuia	F	
350492/	$1,461 \times 10^{-6}$	
350246/	$1,462 \times 10^{-6}$	$\bar{x}_T = 1,455 \times 10^{-6}$
354694,	$1,443 \times 10^{-6}$	

\* vérification des témoins

$$\frac{T_2}{T_1} \times 100 = \frac{1,455 \times 10^{-6}}{1,464 \times 10^{-6}} = 99,4\% \text{ témoins corrects acceptés.}$$

\* moyenne des facteurs de calibration:

$$\overline{TF} = \frac{1,455 \times 10^{-6} + 1,464 \times 10^{-6}}{2} = 1,460 \times 10^{-6}$$

\* dosage du fénofibrate

~~dans chaque préparation (voir procédure pour formule écrite)~~

~~$$375 \times \frac{TF \times A_1 \times 111}{PE} = 694,4 \text{ mg.}$$~~

pour 14 kg A

~~$$\frac{375 \times 1,460 \times 10^{-6} \times 144161 \times 694,4}{351,4} =$$~~

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pour 14 kg A  $\frac{375 \times 1,460 \times 10^{-6} \times 144161 \times 694,4}{351,4} = 155,97 \text{ mg/comprimé}$   
(250,1 + 101,3)

pour 14 kg B  $\frac{375 \times 1,460 \times 10^{-6} \times 176800 \times 694,4}{350,0} = 192,05 \text{ mg/comprimé}$

pour 18 kg A.  $\frac{375 \times 1,460 \times 10^{-6} \times 142054 \times 694,4}{351,1} = 152,74 \text{ mg/comprimé}$

Pas d'injection du 18 kg B.

RCOB.07.97

qu'ils aient une densité de 18 kg ou de 14 kg, les comprimés  
provenant du même mélange on peut donc faire la moyenne  
de ces résultats :

$$\frac{155,97 + 192,05 + 152,74}{3} = 166,92 \text{ mg/comprimé.}$$

Lot 2394/01RG soit 167 mg/comprimé.

noter la valeur importante de 192,05 mg. PC03.07.97  
Les chromatogrammes correspondants sont archivés dans l'annexe 2 -

FOURNIER 1001594

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LOT RG 239610.1  
durée 18 hg.

1. Préparation du milieu de dissolution

Voir page 92

2. Pesée du milieu de dissolution Bal. GAL 111

1 Ltr LSNr 0,025N → 1001,0 g.

bal	remise à zero	masse LSNr 0,025N	vérificateurs
1	oui	1001,1 g	AE 8/4/97. M ob.ch.97
2	oui	1001,1 g	AE 8/4/97. M ob.ch.97
3	oui	1001,0 g	AE 8/4/97. M ob.ch.97
4	oui	1001,0 g	AE 8/4/97. M ob.ch.97
5	oui	1001,1 g	AE 8/4/97 M ob.ch.97
6	oui	1001,1 g	AE 8/4/97. M ob.ch.97

conditions

dissolubilité

GAL 001

T° 37°C ± 0,5

⊙ 75 TPN

AE 8/4/97 M ob.ch.97

AE 8/4/97. M ob.ch.97

3. Pesée des comprimés Bal GAL 205

FOURNIER 1001595  
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 Protective Order

08.04.97 10:16:41  
 Code 2396.01-18

ID 1

0.0 mg

697.3 mg

ID 2

0.0 mg

691.0 mg

ID 3

0.0 mg

695.4 mg

ID 4

0.0 mg

697.3 mg

ID 5

0.0 mg

698.5 mg

ID 6

0.0 mg

696.3 mg

M

REC 07.97



UV GAL 233 - 290nm - cellule 2mm - ChroMo GAL 233<sup>105</sup>

LF 178 TER LOT RG 2396/01 18 KG

04-07-1997 22:57

			Lambda	No.	Valeur_E			
						290.0	$T_{31}$	31 1.2668
1	290.0	1	0.0000	1	0.0003	290.0	$T_{32}$	32 1.2282
2	290.0	2	0.0038	2	0.0002	290.0		33 1.2796
3	290.0	3	0.0001	3	0.0004	290.0		34 1.2527
						290.0		35 1.2689
						290.0		36 1.2545
1	AZ Air / Ach					290.0	$T_{37}$	37 1.2841
						290.0	$T_{38}$	38 1.3173
2	LSNec / LSNec					290.0		39 1.3401
						290.0		40 1.2943
3	A7 1.81. 11.011.					290.0		41 1.2827

h. Lecture

UV GAL 233 - 290nm. cellule 2mm - chrono GAL 123<sup>105</sup>

LF 178 TER LOT RG 2396/01 18 KG

04-07-1997 22:57

	Lambda	No.	Valeur_E			
1	290.0	1	0.0000_1	290.0	T <sub>0</sub>	1 0.0003_1
2	290.0	2	0.0038_1	290.0		2 0.0002_1
3	290.0	3	0.0001_1	290.0		3 0.0004_1
				290.0		4 -0.0007_1
				290.0		5 -0.0003_1
				290.0		6 -0.0008_1
				290.0	T <sub>5</sub>	7 0.3156_1
				290.0		8 0.2900_1
				290.0		9 0.3205_1
				290.0		10 0.3238_1
				290.0		11 0.3137_1
				290.0		12 0.2897_1
				290.0	T <sub>10</sub>	13 0.8292_1
				290.0		14 0.7521_1
				290.0		15 0.8017_1
				290.0		16 0.7868_1
				290.0		17 0.7618_1
				290.0		18 0.7518_1
				290.0	T <sub>15</sub>	19 1.0324_1
				290.0		20 0.8885_1
				290.0		21 1.0791_1
				290.0		22 1.0778_1
				290.0		23 0.8887_1
				290.0		24 1.0141_1
				290.0	T <sub>20</sub>	25 1.1840_1
				290.0		26 1.1833_1
				290.0		27 1.2098_1
				290.0		28 1.1462_1
				290.0		29 1.1858_1
				290.0		30 1.1769_1
				290.0	T <sub>30</sub>	31 1.2668_1
				290.0		32 1.2282_1
				290.0		33 1.2796_1
				290.0		34 1.2527_1
				290.0		35 1.2689_1
				290.0		36 1.2545_1
				290.0	T <sub>35</sub>	37 1.2841_1
				290.0		38 1.3173_1
				290.0		39 1.3401_1
				290.0		40 1.2943_1
				290.0		41 1.3367_1
				290.0		42 1.2740_1
				290.0	T <sub>40</sub>	43 1.3163_1
				290.0		44 1.3455_1
				290.0		45 1.3272_1
				290.0		46 1.3301_1
				290.0		47 1.3408_1
				290.0		48 1.3574_1
				290.0	T <sub>45</sub>	49 1.3327_1
				290.0		50 1.3440_1
				290.0		51 1.3363_1
				290.0		52 1.3355_1
				290.0		53 1.3694_1
				290.0		54 1.3659_1
				290.0	T <sub>50</sub>	55 1.3791_1
				290.0		56 1.4066_1
				290.0		57 1.3854_1
				290.0		58 1.3586_1
				290.0		59 1.3996_1
				290.0		60 1.3930_1

- 1 AZ Air / Ach
- 2 LSNce / LSNce
- 3 AZ LSNce / LSNce

## DISSOLUTION

m:\commun\gl\q\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 08/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2396/01 à 18 KG  
N° CAHIER : LF 178 TER n°1 p 105  
FICHIER : M:\commun\gl\q\donnbases\LF178ter\dissolution\lot 2396RG01 18 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
697,30	691,00	695,40	697,30	698,50	696,30
160,67	159,22	160,23	160,67	160,94	160,44

masse de la prise d'essai  
quantité de principe actif

Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,316	0,29	0,321	0,324	0,314	0,29
10	1000	0,829	0,752	0,802	0,787	0,762	0,752
15	1000	1,032	0,889	1,079	1,078	0,889	1,014
20	1000	1,184	1,183	1,21	1,146	1,186	1,177
30	1000	1,267	1,228	1,28	1,253	1,269	1,255
40	1000	1,284	1,317	1,34	1,294	1,337	1,274

FOURNIER 1001597

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AC 03.07.97

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 08/04/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2

TITRE : LF 178 TER RG 2396/01 à 18 KG  
 N° CAHIER : LF 178 TER n°1 p 105  
 FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2396RG01 18 kg  
 ELUANT : LSNa 0,025 M  
 AGITATION : 75 TPM

FOURNIER 1001598

## PREPARATION DES ECHANTILLONS

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 Subject to  
 Protective Order

masse théorique 694,4  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	697,30	691,00	695,40	697,30	698,50	696,30
quantité de principe actif	160,67	159,22	160,23	160,67	160,94	160,44

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

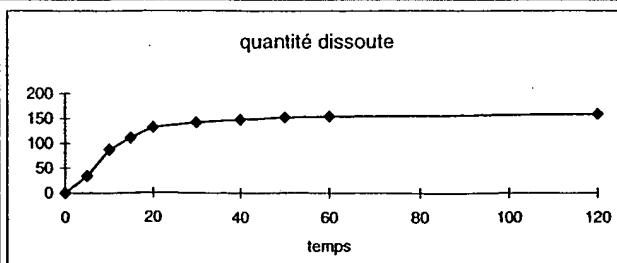
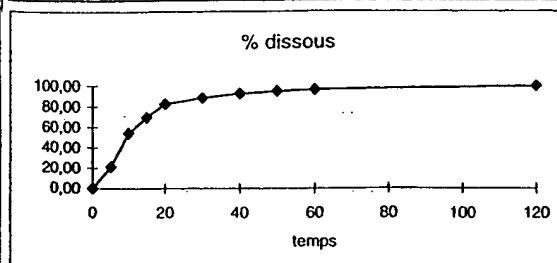
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,316	0,29	0,321	0,324	0,314	0,29
10	1000	0,829	0,752	0,802	0,787	0,762	0,752
15	1000	1,032	0,889	1,079	1,078	0,889	1,014
20	1000	1,184	1,183	1,21	1,146	1,186	1,177
30	1000	1,267	1,228	1,28	1,253	1,269	1,255
40	1000	1,284	1,317	1,34	1,294	1,337	1,274
50	1000	1,316	1,346	1,327	1,33	1,341	1,357
60	1000	1,333	1,344	1,336	1,336	1,369	1,366
120	1000	1,379	1,407	1,385	1,359	1,4	1,393

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	21,42	21,85	20,24	22,26	22,41	21,68	20,08	1,01	4,72
10,0	54,20	57,44	52,58	55,73	54,54	52,71	52,18	2,09	3,86
15,0	69,44	71,76	62,40	75,21	74,93	61,75	70,59	5,98	8,61
20,0	82,55	82,63	83,23	84,67	80,01	82,56	82,23	1,52	1,84
30,0	88,34	88,78	88,78	89,94	87,81	88,70	88,03	1,07	1,21
40,0	92,18	90,40	93,42	94,55	91,07	93,83	89,78	2,00	2,17
50,0	94,60	93,05	95,91	94,11	94,01	94,57	95,97	1,15	1,21
60,0	95,84	94,68	96,24	95,20	94,89	96,96	97,07	1,06	1,10
120,0	99,07	98,33	101,10	99,06	96,94	99,57	99,41	1,39	1,40
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	34,35	35,11	32,22	35,67	36,00	34,89	32,22	1,70	4,94
10,0	86,91	92,29	83,72	89,29	87,62	84,84	83,72	3,46	3,98
15,0	111,36	115,30	99,36	120,51	120,40	99,38	113,25	9,72	8,73
20,0	132,38	132,77	132,52	135,67	128,55	132,87	131,92	2,28	1,73
30,0	141,67	142,65	138,17	144,12	141,08	142,75	141,24	2,04	1,44
40,0	147,81	145,24	148,75	151,50	146,33	151,01	144,05	3,09	2,09
50,0	151,70	149,51	152,70	150,80	151,05	152,20	153,98	1,58	1,04
60,0	153,69	152,13	153,23	152,53	152,45	156,05	155,73	1,75	1,14
120,0	158,86	157,98	160,97	158,72	155,75	160,26	159,49	1,86	1,17
0,0									
0,0									
0,0									



8/4/97

Dissolution des comprimés enrobés OPADRY 07  
Lot 2393/01 RG stockés 15 jours à  
75% HR.

1. Préparation du LSNa 0,025M

masse molaire du LSNa = 283,4 g.

pesée eau : Tare = 2,800 kg de 8/4/97 à 08/04/97

brut = 22,590 kg. de 8/4/97 à 08/04/97

net = 22,590 - 2,800 = 19,790 kg.

soit  $19,790 \times 283,4 \times 0,025 = 143$  g de LSNa à peser.

Tare = 161,6 g de 8/4/97. à 08/04/97

brut = 204,6 g de 8/4/97 M. ob. ch. 97

net = 143,0 g.

2. Pesée du milieu de dissolution gel III.

1 litre de LSNa 0,025M = 1001,0 g.

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lots	gros	Pesée	Signature
1	oui	1001,0 g.	M. ob. ch. 97 de 8/4/97
2	oui	1001,0 g.	M. ob. ch. 97 de 8/4/97
3	oui	1001,0 g.	M. ob. ch. 97 de 8/4/97
4	oui	1001,0 g.	M. ob. ch. 97 de 8/4/97
5	oui	1001,0 g.	M. ob. ch. 97 de 8/4/97
6	oui	1001,0 g.	M. ob. ch. 97 de 8/4/97.

PO 02/06/97  
PC 03.07.97

### 3. Pesée des comprimés galés.

08.04.97	13:34:03	2393.01	1	0.0 mg
Code	ID		2	475.9 mg
	ID		3	0.0 mg
	ID		4	481.8 mg
	ID		5	0.0 mg
	ID		6	467.0 mg
	ID		7	483.0 mg
	ID		8	0.0 mg
	ID		9	474.2 mg
	ID		10	0.0 mg
	ID		11	476.7 mg

### 4. Conditions de dissolution

dissolvant: Pistolet gal 103. équipé de pelotes tournantes.

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  du 08/04/97 au 8/4/97

$\gamma = 75 \text{ RPM}$  du 08/04/97 au 8/4/97.

S. Lecture

spectrophotométrie KONTRON 930 gal 103.

cuvettes de 2 mm de trajet optique.

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LF 178TER ENROBE OPADRY DY 15 JOURS A 75HR

08-04-1996 13:40

Lambda	No.	Valeur_E			
290.0	1	-0.0001_1	290.0	22	0.7360_1
290.0	2	0.0042_1	290.0	23	0.7550_1
290.0	3	0.0001_1	290.0	24	0.7454_1
290.0	4	0.0003_1	290.0	25	0.7662_1
290.0	5	0.0005_1	290.0	26	0.7533_1
290.0	6	0.0005_1	290.0	27	0.7465_1
290.0	7	-0.0001_1	290.0	28	0.7918_1
290.0	8	-0.0003_1	290.0	29	0.8113_1
290.0	9	-0.0003_1	290.0	30	0.7894_1
290.0	10	0.4479_1	290.0	31	0.8110_1
290.0	11	0.4029_1	290.0	32	0.8090_1
290.0	12	0.4869_1	290.0	33	0.7977_1
290.0	13	0.4048_1	290.0	34	0.8426_1
290.0	14	0.4466_1	290.0	35	0.8574_1
290.0	15	0.4413_1	290.0	36	0.8334_1
290.0	16	0.6514_1	290.0	37	0.8734_1
290.0	17	0.6490_1	290.0	38	0.8504_1
290.0	18	0.6692_1	290.0	39	0.8296_1
290.0	19	0.6553_1	290.0	40	0.8588_1
290.0	20	0.6585_1	290.0	41	0.8642_1
290.0	21	0.6509_1	290.0	42	0.8528_1
			290.0	43	0.8856_1
			290.0	44	0.8563_1
			290.0	45	0.8473_1
			290.0	46	0.8501_1
			290.0	47	0.8651_1
			290.0	48	0.8759_1
			290.0	49	0.7891_1
			290.0	50	0.8747_1
			290.0	51	0.8577_1
			290.0	52	0.8862_1
			290.0	53	0.8618_1
			290.0	54	0.8763_1
			290.0	55	0.8610_1
			290.0	56	0.8971_1
			290.0	57	0.8755_1
			290.0	58	0.8802_1

02/06/97

RC 03.07.97

# DISSOLUTION

m:\commun\gl\ql\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
 DATE : 08/04/97  
 APPAREIL : gal 103 108  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2mm

TITRE : comprimés enrobés OPADRY OY lot 2393/01RG 15 jours à 75% HR  
 N° CAHIER : LF 178ter dissolution page 106  
 FICHER : m:\commun\gl\ql\donnbase\lf178ter\dissolution\lot 2393rg01 75hr  
 ELUANT : LSNa 0,025M  
 AGITATION : 75 TPM

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## PREPARATION DES ECHANTILLONS

masse théorique	100
dosage théorique	100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	100,00	100,00	100,00	100,00	100,00	100,00
quantité de principe actif	100,00	100,00	100,00	100,00	100,00	100,00

Témoin 100mg/ 0,900

## SUIVI DE LA DISSOLUTION

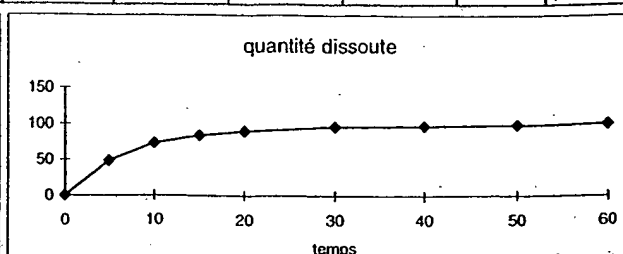
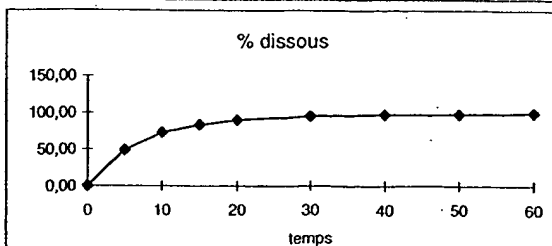
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,448	0,403	0,487	0,405	0,447	0,441
10	1000	0,651	0,649	0,669	0,655	0,659	0,651
15	1000	0,736	0,755	0,745	0,766	0,753	0,747
20	1000	0,792	0,811	0,789	0,811	0,809	0,798
30	1000	0,843	0,857	0,833	0,873	0,85	0,83
40	1000	0,859	0,864	0,853	0,886	0,856	0,847
50	1000	0,85	0,865	0,876		0,875	0,858
60	1000	0,886	0,862	0,876	0,861	0,897	0,876

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	48,72	49,78	44,78	54,11	45,00	49,67	49,00	3,48	7,14
10,0	73,10	72,58	72,34	74,60	73,00	73,47	72,58	0,84	1,15
15,0	83,98	82,39	84,47	83,42	85,70	84,28	83,61	1,12	1,33
20,0	90,10	89,02	91,12	88,72	91,13	90,92	89,69	1,09	1,21
30,0	95,66	95,13	96,68	94,05	98,47	95,93	93,69	1,77	1,85
40,0	97,59	97,37	97,93	96,74	100,39	97,07	96,04	1,51	1,55
50,0	98,50	96,85	98,52	99,76		99,65	97,73	1,25	1,27
60,0	100,19	101,32	98,67	100,25	98,11	102,58	100,21	1,65	1,65
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	48,72	49,78	44,78	54,11	45,00	49,67	49,00	3,48	7,14
10,0	73,10	72,58	72,34	74,60	73,00	73,47	72,58	0,84	1,15
15,0	83,98	82,39	84,47	83,42	85,70	84,28	83,61	1,12	1,33
20,0	90,10	89,02	91,12	88,72	91,13	90,92	89,69	1,09	1,21
30,0	95,66	95,13	96,68	94,05	98,47	95,93	93,69	1,77	1,85
40,0	97,59	97,37	97,93	96,74	100,39	97,07	96,04	1,51	1,55
50,0	98,50	96,85	98,52	99,76		99,65	97,73	1,25	1,27
60,0	100,19	101,32	98,67	100,25	98,11	102,58	100,21	1,65	1,65
0,0									
0,0									
0,0									
0,0									



durée 14 hq.1. Préparation du milieu de dissolution

Voir page 106

2. Pesée du milieu de dissolution Bal GAL-M

1 Ltr LNa 0,025N → 1001,0 g.

bal	mise à zero	masse LNa 0,025N	vérification
1	oui.	1001,0 g.	Re- 8/4/97. M. Ob. Ch. 97
2	oui.	1001,1 g.	Re- 8/4/97 M. Ob. Ch. 97
3	oui.	1001,0 g.	Re- 8/4/97. M. Ob. Ch. 97
4	oui.	1001,0 g.	Re- 8/4/97. M. Ob. Ch. 97
5	oui.	1001,0 g.	Re- 8/4/97. M. Ob. Ch. 97
6	oui.	1001,1 g.	Re- 8/4/97. M. Ob. Ch. 97

Conditions

Dissoluté

T° 37°C ± 0,5 M. Ob. Ch. 97 Re- 20/4/97

GAL 021

O 75 TPN M. Ob. Ch. 97 Re- 20/4/97

3. Pesée des comprimés Bal GAL 205

08.04.97 Code 2396.01-14	13:38:35	1	2	3	4	5	6
ID		0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
		691.4 mg	694.6 mg	691.5 mg	694.7 mg	689.4 mg	686.2 mg
ID							
ID							
ID							
ID							
ID							

M

FOURNIER 1001602

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Re 030797

110

h. Lecture

UV GAL 233 - 280nm - cellule 2mm - chrono GAL

123

LF 178 TER LOT RG 2396/01 14 KG

04-08-1997 16:02

			Lambda	No.	Valeur_E				
						290.0	T <sub>0</sub>	31	1.2555
1	290.0	1	-0.0000	1	0.0001	290.0	T <sub>30</sub>	32	1.2655
2	290.0	2	0.0029	2	0.0006	290.0		33	1.2775
3	290.0	3	0.0000	3	-0.0002	290.0		34	1.3067
						290.0		35	1.2469
						290.0		36	1.2617
						290.0	T <sub>5</sub>	37	1.3103
						290.0		38	1.3117

A2 A4 / A4

FOURNIER 1001603  
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RV 03.07.04



# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR: D.LECRIT  
 DATE: 08/04/97  
 APPAREIL: GAL 233 GAL 091  
 LONGUEUR D'ONDE: 290 nm  
 CUVE en mm: 2

TITRE: LF 178 TER RG 2396/01 à 14 KG  
 N° CAHIER: 178 TER n°1 p 110  
 FICHIER: M:\commun\glnq\donbase\LF178ter\dissolution\lot 2396RG01 14 kg  
 ELUANT: LSNa 0,025 M  
 AGITATION: 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 694,4  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	691,40	694,60	691,50	694,70	689,40	686,20
quantité de principe actif	159,31	160,05	159,33	160,07	158,85	158,11

Témoin 100mg/l 0,900

## SUIVRE LA DISSOLUTION

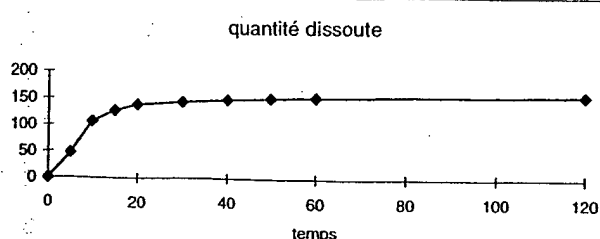
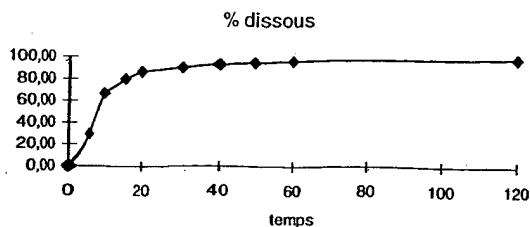
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,444	0,408	0,436	0,352	0,433	0,471
10	1000	0,976	0,973	0,961	0,949	0,949	0,926
15	1000	1,148	1,149	1,158	1,108	1,122	1,14
20	1000	1,233	1,229	1,24	1,244	1,203	1,227
30	1000	1,256	1,266	1,278	1,307	1,247	1,262
40	1000	1,31	1,312	1,31	1,32	1,281	1,292
50	1000	1,3	1,339	1,322	1,333	1,334	1,313
60	1000	1,33	1,326	1,349	1,365	1,34	1,325
120	1000	1,363	1,368	1,37	1,373	1,358	1,36

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	29,59	30,97	28,33	30,40	24,43	30,29	33,10	2,95	9,97
10,0	66,81	68,23	67,69	67,17	66,00	66,53	65,24	1,11	1,66
15,0	79,83	80,56	80,25	81,24	77,36	78,97	80,60	1,42	1,78
20,0	86,63	86,89	86,20	87,36	87,19	85,02	87,12	0,89	1,02
30,0	89,85	88,93	89,20	90,45	91,99	88,52	90,01	1,27	1,41
40,0	92,72	93,13	92,83	93,12	93,35	91,34	92,56	0,73	0,79
50,0	94,53	92,89	95,16	94,42	94,71	95,49	94,49	0,90	0,95
60,0	96,08	95,44	94,72	96,76	97,39	96,38	95,79	0,96	1,00
120,0	98,37	98,20	98,10	98,69	98,42	98,11	98,72	0,28	0,29
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	47,11	49,33	45,33	48,44	39,11	48,11	52,33	4,52	9,59
10,0	106,42	108,69	108,34	107,02	105,64	105,69	103,15	2,05	1,93
15,0	127,16	128,34	128,43	129,44	123,83	125,43	127,44	2,11	1,66
20,0	137,99	138,43	137,96	139,20	139,56	135,06	137,74	1,60	1,16
30,0	143,12	141,67	142,76	144,11	147,25	140,62	142,31	2,33	1,63
40,0	147,69	148,37	148,57	148,37	149,42	145,09	146,35	1,63	1,10
50,0	150,57	147,98	152,30	150,44	151,60	151,69	149,40	1,64	1,09
60,0	153,04	152,04	151,60	154,17	155,90	153,09	151,46	1,73	1,13
120,0	156,69	156,44	157,00	157,25	157,54	155,84	156,09	0,68	0,43
0,0									
0,0									
0,0									



h. Lecture

UV GAL 233 - 290nm - cellule 2mm - chrono GAL

123

LF 178 TER LOT RG 2396/01 14 KG

04-08-1997 16:02

M

1 290.0 1 -0.0000\_1  
2 290.0 2 0.0029\_1  
3 290.0 3 0.0000\_1

1 AZ A<sub>1</sub> / A<sub>2</sub>

2 LSN<sub>1</sub> / LSN<sub>2</sub>

3 AZ LSN<sub>1</sub> / LSN<sub>2</sub>

Lambda No. Valeur\_E

290.0	T <sub>0</sub>	1	0.0001_1	290.0	T <sub>35</sub>	31	1.2555_1
290.0		2	0.0006_1	290.0		32	1.2655_1
290.0		3	-0.0002_1	290.0		33	1.2775_1
290.0		4	-0.0002_1	290.0		34	1.3067_1
290.0		5	0.0002_1	290.0		35	1.2469_1
290.0		6	-0.0002_1	290.0		36	1.2617_1
290.0	T <sub>5</sub>	7	0.4444_1	290.0	T <sub>40</sub>	37	1.3103_1
290.0		8	0.4076_1	290.0		38	1.3117_1
290.0		9	0.4362_1	290.0		39	1.3095_1
290.0		10	0.3517_1	290.0		40	1.3200_1
290.0		11	0.4333_1	290.0		41	1.2811_1
290.0		12	0.4705_1	290.0		42	1.2923_1
290.0	T <sub>10</sub>	13	0.9755_1	290.0	T <sub>45</sub>	43	1.3004_1
290.0		14	0.9733_1	290.0		44	1.3385_1
290.0		15	0.9609_1	290.0		45	1.3224_1
290.0		16	0.9487_1	290.0		46	1.3334_1
290.0		17	0.9490_1	290.0		47	1.3343_1
290.0		18	0.9264_1	290.0		48	1.3129_1
290.0	T <sub>15</sub>	19	1.1484_1	290.0	T <sub>50</sub>	49	1.3302_1
290.0		20	1.1492_1	290.0		50	1.3264_1
290.0		21	1.1581_1	290.0		51	1.3494_1
290.0		22	1.1083_1	290.0		52	1.3651_1
290.0		23	1.1221_1	290.0		53	1.3395_1
290.0		24	1.1396_1	290.0		54	1.3254_1
290.0	T <sub>20</sub>	25	1.2330_1	290.0	T <sub>55</sub>	55	1.3312_1
290.0		26	1.2293_1	290.0		56	1.3628_1
290.0		27	1.2397_1	290.0		57	1.3675_1
290.0		28	1.2438_1	290.0		58	1.3702_1
290.0		29	1.2025_1				
290.0		30	1.2266_1				
				290.0		59	1.3730_1
				290.0		60	1.3581_1
				290.0		61	1.3600_1

↳ Erreur de renvoi M de ch 97.

## DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 08/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2396/01 à 14 KG  
N° CAHIER : 178 TER n°1 p 110  
FICHIER : M:\commun\glnq\donbase\LF178ter\dissolution\lot 2396RG01 14 kg  
ELUANT : LSN a 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	691,40	694,60	691,50	694,70	689,40	686,20
quantité de principe actif	159,31	160,05	159,33	160,07	158,85	158,11

Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,444	0,408	0,436	0,352	0,433	0,471
10	1000	0,444	0,408	0,436	0,352	0,433	0,471

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AL 03.07.97

Dosage du fenofibrate dans  
les mélanges avant compression.

lot 2394/03 RG

lot 2396/03 RG.

Dosage effectué par HPLC selon procédure 02 DAP004-06/05.

conditions chromatographiques

voir p.99.

1. Préparation des solutions témoins.

balance DAP nos.

1.1 Solutions témoins mes. (en double).

introduire 25,00 mg environ exactement connue de fenofibrate  
au 1241 dans fiole jaugée de 50 ml.

\*\*\* 08/04/97 - 08:56 \*\*\*

Echantillon: FENOFIBRATE  
T1  
No controle: ARR 1241  
Operateur : AG

Poids tare : 37073.13 g

Poids brut : 37098.52 g

POIDS NET : 25.39 g

\*\*\* 08/04/97 - 08:59 \*\*\*

Echantillon: FENOFIBRATE  
T2  
No controle: ARR 1241  
Operateur : AG

Poids tare : 37365.84 g

Poids brut : 37393.42 g

POIDS NET : 27.58 g

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L'éprouve imprimée des g alors que j'ai pesé des mg.  
ajouter 5 ml de THF.

qsp ACW.  
agitation magnétique pdt 30 minutes.

08 03 07.97

soient SM1 et SM2

## 1.2. Solutions rénovés de travail.

dilués au 2/20<sup>e</sup> dans la phase mobile.

SM1  $\bar{a} = 0,5078 \mu\text{g}/10 \mu\text{L}$

SM2  $\bar{a} = 0,5516 \mu\text{g}/10 \mu\text{L}$

## 2. Préparation des solutions essais balance Dap 3.

### 2.1 solutions essais mères

à faire en double.

Peser exactement environ 350 mg de mélange dans fiole de 150 mL.

\*\*\* 08/04/97 - 09:28 \*\*\*

Echantillon: LF 178TER  
No controle: 2394RG01 1  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 19456.64 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 19885.45 g

POIDS NET : 348.81 g

\*\*\* 08/04/97 - 09:31 \*\*\*

Echantillon: LF 178TER  
No controle: 2394RG01 2  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 18884.78 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 19236.38 g

POIDS NET : 351.60 g

\*\*\* 08/04/97 - 09:38 \*\*\*

Echantillon: LF 178TER  
No controle: 2396RG01  
Operateur : AG 4

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 19344.91 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 19695.50 g

POIDS NET : 350.59 g

\*\*\* 08/04/97 - 09:34 \*\*\*

Echantillon: LF 178TER  
No controle: 2396RG01 3  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 20139.44 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 20486.63 g

POIDS NET : 347.19 g

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l'éprouve en prime des g alors que j'ai pesé des mg.

Re 03.07.97

ajouter 15 ml de THF + 7,5 ml de phase mobile.

agitation magnétique pour 45 min

qsp avec ACN.

agitation magnétique pour 45 min

## 2.2. Solutions essai de travail.

diluer chaque solution essai de travail, même au 1/25<sup>e</sup>

qsp avec phase mobile

## 3. Injections et résultats.

les chromatogrammes sont dans l'annexe 2.

\* injecter 5 fois T1 et calculer le facteur de calibration F.

aire	$F = \frac{CF}{Aire}$ — conc en fois en $\mu g / 10 \mu l$	
348693,	$1,456 \times 10^{-6}$	$\bar{x} = 1,465 \times 10^{-6}$ $E_c = 5,26 \times 10^{-3}$ $\omega = 0,4\%$  moyenne des 3 dernières injections $F_{T1} = 1,468 \times 10^{-6}$
348287,	$1,466 \times 10^{-6}$	
345991,	$1,468 \times 10^{-6}$	
346094,	$1,467 \times 10^{-6}$	
<del>348</del> 345776,	$1,469 \times 10^{-6}$	

\* calcul du facteur de calibration de T2.

aire	F
368962,	$1,495 \times 10^{-6}$
368570,	$1,497 \times 10^{-6}$
368193,	$1,498 \times 10^{-6}$

$$F_{T2} = 1,497 \times 10^{-6}$$

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RC03.07.97

\* vérification des rétrovis

$$\frac{F_{T2} \times 100}{F_{T1}} = \frac{1,497 \times 10^{-6} \times 100}{1,468 \times 10^{-6}} = 102,0\% \text{ rétrovis acceptés}$$

\* moyenne des facteurs de calibration

$$MF = \frac{F_{T1} + F_{T2}}{2} = \frac{1,497 \times 10^{-6} + 1,468 \times 10^{-6}}{2}$$

$$MF = 1,483 \times 10^{-6}$$

\* dosage du fénofibrate par comptage

voir procédure pour formule édatée

$$375 \times \frac{MF \times A_{\text{ave}} \times 694,4}{PE}$$

solution 2394/01 RG 1.

$$\frac{375 \times 1,483 \times 10^{-6} \times 140998 \times 694,4}{348,81} = 156,10 \text{ mg}$$

solution 2394/01 RG 2

$$\frac{375 \times 1,483 \times 10^{-6} \times 140168 \times 694,4}{351,60} = 153,95 \text{ mg}$$

solution 2396/01 RG 3

$$\frac{375 \times 1,483 \times 10^{-6} \times 139400 \times 694,4}{350,59} = 153,55 \text{ mg}$$

solution 2396/01 RG 4

$$\frac{375 \times 1,483 \times 10^{-6} \times 135583 \times 694,4}{347,19} = 150,81 \text{ mg}$$

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R. 03 0791

lot 2394/cu RG = 155 mg de fenofibrate / comprimé

lot 2396/o.1 RG = 152 mg de fenofibrate / comprimé

RC 03.07.97

FOURNIER 1001610

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LOT RG 2397/01  
durée 14 hg.

1. Préparation du milieu de dissolution

Voir page 106

2. Remplissage des bts de dissolution Bal GAL III

1 litre LSNu 0,025N  $\rightarrow$  1001,0 g.

btl	remise à zéro	masse LSNu 0,025N	Vérifications
1	oui	1001,0 g.	82 03/04/97 M. O. S. Ch. 97
2	oui	1002,1 g.	82 03/04/97 M. O. S. Ch. 97
3	oui	1002,0 g.	82 03/04/97 M. O. S. Ch. 97
4	oui	1002,0 g.	82 03/04/97 M. O. S. Ch. 97
5	oui	1002,0 g.	82 03/04/97 M. O. S. Ch. 97
6	oui	1001,0 g.	82 03/04/97 M. O. S. Ch. 97

Conditions

Dissolution  
 GAL 091

7: 37°C  $\pm$  0,5 82 03/04/97 M. O. S. Ch. 97  
 8 75 TPN 82 03/04/97 M. O. S. Ch. 97

3. Pesée des comprimés Bal GAL 205

09.04.97 10:01:45  
 Code 2397.01-14

ID	1	0.0 mg
ID	2	686.8 mg
ID	3	0.0 mg
ID	3	691.3 mg
ID	4	0.0 mg
ID	4	692.2 mg
ID	5	0.0 mg
ID	5	690.7 mg
ID	6	0.0 mg
ID	6	694.5 mg
ID		0.0 mg
ID		689.8 mg

M

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 Protective Order

REC 03.07.97





h. lecture

Spectro GAL 233

chronométré GAL 233

117

LF 178 TER LOT RG 2397/01 14 KG

04-09-1997 12:28

Lambda	No.	Valeur_E
290.0	1	-0.0000_1
290.0	2	-0.0026_1
290.0	3	0.0001_1

1 AZ A<sub>1</sub>/A<sub>2</sub>  
2 LSNa/LSNa  
3 AZ LSNa/LSNa

Lambda	No.	Valeur_E
290.0	1	0.0008_1
290.0	2	0.0008_1
290.0	3	0.0007_1
290.0	4	0.0009_1
290.0	5	0.0032_1
290.0	6	0.0019_1
290.0	7	0.5446_1
290.0	8	0.5130_1
290.0	9	0.4819_1
290.0	10	0.5826_1
290.0	11	0.4591_1
290.0	12	0.6254_1
290.0	13	1.0050_1
290.0	14	1.0024_1
290.0	15	1.0107_1
290.0	16	1.0189_1
290.0	17	0.9647_1
290.0	18	1.0075_1
290.0	19	1.1528_1
290.0	20	1.1599_1
290.0	21	1.1662_1
290.0	22	1.1719_1
290.0	23	1.1537_1
290.0	24	1.1429_1
290.0	25	1.2428_1
290.0	26	1.2449_1
290.0	27	1.2508_1
290.0	28	1.2186_1
290.0	29	1.2528_1
290.0	30	1.2297_1

290.0	31	1.3016_1
290.0	32	1.2830_1
290.0	33	1.3091_1
290.0	34	1.2904_1
290.0	35	1.3245_1
290.0	36	1.2777_1
290.0	37	1.3313_1
290.0	38	1.3263_1
290.0	39	1.3241_1
290.0	40	1.3126_1
290.0	41	1.3600_1
290.0	42	1.3332_1
290.0	43	1.3491_1
290.0	44	1.3534_1
290.0	45	1.3386_1
290.0	46	1.3306_1
290.0	47	1.3623_1
290.0	48	1.3603_1
290.0	49	1.3467_1
290.0	50	1.3725_1
290.0	51	1.3518_1
290.0	52	1.3465_1
290.0	53	1.3777_1
290.0	54	1.3645_1
290.0	55	1.3822_1
290.0	56	1.3865_1
290.0	57	1.3596_1
290.0	58	1.3530_1
290.0	59	1.4105_1
290.0	60	1.4051_1

DISSOLUTION

m:\commun\glnq\traitdon\distern5  
date édition: le 06/02/97

CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 09/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2397/01 à 14 KG  
N° CAHIER : 178 TER n°1 p 117  
FICHIER : M:\commun\glnq\donnbases\Lf178ter\dissolution\lot 2397RG01 14 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

FOURNIER 1001613

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PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	686,80	691,30	692,20	690,70	694,50	689,80
quantité de principe actif	158,25	159,29	159,49	159,15	160,02	158,94

Témoin 100mg/l 0,900

SUIVI DE LA DISSOLUTION

volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
5	1000	0,545	0,513	0,482	0,583	0,459	0,625
10	1000	1,005	1,002	1,011	1,019	0,965	1,008
15	1000	1,153	1,16	1,166	1,172	1,154	1,143
20	1000	1,243	1,245	1,251	1,219	1,253	1,23
30	1000	1,302	1,283	1,309	1,29	1,325	1,278
40	1000	1,331	1,326	1,324	1,313	1,36	1,333
50	1000	1,349	1,353	1,339	1,331	1,362	1,36
60	1000	1,347	1,373	1,352	1,347	1,378	1,365

RC 0307.92

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 09/04/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVES en mm : 2

TITRE : LF 178 TER RG 2397/01 à 14 KG  
 N° CAHIER : 178 TER n°1 p 117  
 FICHER : M:\commun\glnq\donnbase\L178ter\dissolution\lot 2397RG01 14 kg  
 ELUANT : LSNa 0,025 M  
 AGITATION : 75 TPM

FOURNIER 1001614

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## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	686,80	691,30	692,20	690,70	694,50	689,80
quantité de principe actif	158,25	159,29	159,49	159,15	160,02	158,94

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

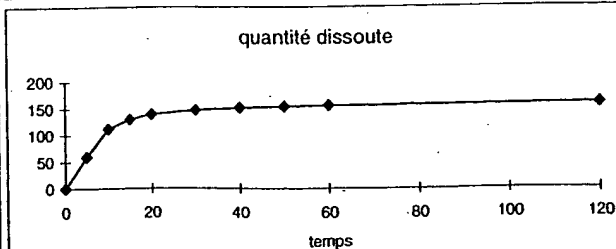
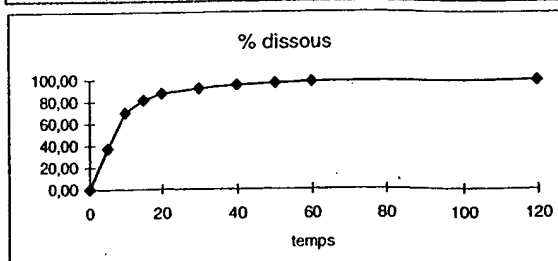
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,545	0,513	0,482	0,583	0,459	0,625
10	1000	1,005	1,002	1,011	1,019	0,965	1,008
15	1000	1,153	1,16	1,166	1,172	1,154	1,143
20	1000	1,243	1,245	1,251	1,219	1,253	1,23
30	1000	1,302	1,283	1,309	1,29	1,325	1,278
40	1000	1,331	1,326	1,324	1,313	1,36	1,333
50	1000	1,349	1,353	1,339	1,331	1,362	1,36
60	1000	1,347	1,373	1,352	1,347	1,378	1,365
120	1000	1,382	1,387	1,36	1,353	1,411	1,405

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	37,32	38,27	35,78	33,58	40,70	31,87	43,69	4,45	11,92
10,0	70,10	70,76	70,07	70,60	71,35	67,16	70,69	1,50	2,13
15,0	81,36	81,50	81,45	81,75	82,38	80,62	80,48	0,71	0,88
20,0	87,50	88,22	87,78	88,08	86,07	87,90	86,96	0,83	0,95
30,0	91,96	92,80	90,86	92,55	91,46	93,33	90,74	1,08	1,18
40,0	94,74	95,30	94,31	94,05	93,51	96,22	95,03	0,97	1,03
50,0	96,45	97,03	96,66	95,56	95,23	96,83	97,39	0,86	0,89
60,0	97,71	97,36	98,52	96,93	96,81	98,42	98,21	0,77	0,79
120,0	99,77	100,29	99,98	97,96	97,70	101,19	101,49	1,60	1,61
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	59,39	60,56	57,00	53,56	64,78	51,00	69,44	6,95	11,71
10,0	111,59	111,97	111,62	112,60	113,55	107,48	112,35	2,12	1,90
15,0	129,52	128,97	129,73	130,39	131,11	129,01	127,91	1,14	0,88
20,0	139,29	139,61	139,82	140,48	136,99	140,65	138,21	1,42	1,02
30,0	146,39	146,86	144,73	147,62	145,55	149,35	144,23	1,93	1,32
40,0	150,81	150,80	150,22	150,01	148,82	153,98	151,05	1,73	1,15
50,0	153,54	153,54	153,96	152,41	151,55	154,95	154,79	1,34	0,87
60,0	155,54	154,07	156,93	154,60	154,07	157,49	156,10	1,50	0,96
120,0	158,82	158,71	159,25	156,24	155,49	161,92	161,30	2,60	1,64
0,0									
0,0									
0,0									



LOT KG 2337/01

discreti ~~th~~ 18 hq.  
M

1. Préparation du milieu de dissolution

• pesée de l'eau Bal GAL 014

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Tare = 2,730 kg M 10.04.97 AR 10/4/97

Brut = 25,080 kg M 10.04.97 AR 10/4/97

Net = 25,080 - 2,730 = 22,350

pour un milieu à 0,025N (PM 286,4 g)

masse de LNa = 22,350 × 286,4 × 0,025 = 161,1 g.

• pesée du LNa Bal GAL 011

Tare = 153,5 g puis R.A.2. M 10.04.97 AR 10/4/97

Net = 161,1 g M 10.04.97 AR 10/4/97 M 10.04.97

2. Remplissage des bts de dissolution Bal GAL 111

1 litre LNa 0,025N → 1001,0 g.

btl	remise à zero	masse LNa 0,025N	Vérificateurs
1	oui	1001,1 g.	SR 10/04/97 M 10.04.97
2	oui	1001,1 g.	SR 10/04/97 M 10.04.97
3	oui	1001,0 g.	SR 10/04/97 M 10.04.97
4	oui	1001,0 g.	SR 10/04/97 M 10.04.97
5	oui	1001,0 g.	SR 10/04/97 M 10.04.97
6	oui	1001,0 g.	SR 10/04/97 M 10.04.97

Conditions

Dissolubilité  
GAL 031

T° = 37°C ± 0,5 AR 10/4/97 M 10.04.97

Θ = 75 TPA AR 10/4/97 M 10.04.97

SR 03.97

10.04.97	09:36:37	1	0.0 mg
Code	2397.01-18	2	692.6 mg
ID		3	0.0 mg
		4	686.3 mg
		5	0.0 mg
		6	695.7 mg
		7	0.0 mg
		8	689.2 mg
		9	0.0 mg
		10	687.6 mg
		11	0.0 mg
		12	691.2 mg

k. Lecture Spectro GAL 233. chromométrie GAL 123

LF 178 TER LOT RG 2397/01 18 KG

04-10-1997 12:28

1 290.0 1 0.0000\_1  
2 290.0 2 0.0030\_1  
3 290.0 3 -0.0001\_1

1. AZ Air/Air

2. LSNa/LSNa

3. AZ LSNa/LSNa

Lambda No. Valeur\_E

290.0	T <sub>0</sub>	1	0.0004_1
290.0		2	0.0008_1
290.0		3	0.0001_1
290.0		4	0.0004_1
290.0		5	0.0015_1
290.0		6	-0.0004_1
290.0	T <sub>5</sub>	7	0.2840_1
290.0		8	0.2967_1
290.0		9	0.2786_1
290.0		10	0.2908_1
290.0		11	0.2936_1
290.0		12	0.2957_1
290.0	T <sub>10</sub>	13	0.8718_1
290.0		14	0.8589_1
290.0		15	0.8411_1
290.0		16	0.8680_1
290.0		17	0.8186_1
290.0		18	0.8124_1
290.0	T <sub>15</sub>	19	1.1019_1
290.0		20	1.0944_1
290.0		21	1.0972_1
290.0		22	1.0608_1
290.0		23	1.0774_1
290.0		24	1.0889_1
290.0	T <sub>20</sub>	25	1.1906_1
290.0		26	1.1830_1
290.0		27	1.2091_1
290.0		28	1.1822_1
290.0		29	1.1775_1
290.0		30	1.1887_1

290.0	T <sub>30</sub>	31	1.2554_1
290.0		32	1.2500_1
290.0		33	1.2820_1
290.0		34	1.2492_1
290.0		35	1.2597_1
290.0		36	1.2481_1
290.0	T <sub>40</sub>	37	1.2948_1
290.0		38	1.2920_1
290.0		39	1.2847_1
290.0		40	1.2971_1
290.0		41	1.2976_1
290.0		42	1.2788_1
290.0	T <sub>50</sub>	43	1.3158_1
290.0		44	1.3202_1
290.0		45	1.3185_1
290.0		46	1.3121_1
290.0		47	1.3229_1
290.0		48	1.3222_1
290.0	T <sub>60</sub>	49	1.3281_1
290.0		50	1.3337_1
290.0		51	1.3431_1
290.0		52	1.3340_1
290.0		53	1.3497_1
290.0		54	1.3467_1
290.0	T <sub>70</sub>	55	1.3607_1
290.0		56	1.3604_1
290.0		57	1.3777_1
290.0		58	1.3618_1
290.0		59	1.3689_1
290.0		60	1.3569_1

FOURNIER 1001616

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RC03.07.97

## DISSOLUTION

m:\commun\glnq\traitdon\diem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 10/04/97  
 APPAREIL : GAL 233 GAL 094  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2

TITRE : LF 178 TER RG 2397/01 à 18 KG  
 N° CAHIER : LF 178 TER n°1 p 120  
 FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2397RG01 18 kg  
 ELUANT : LNa 0,025 M  
 AGITATION : 75 TPM

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## PREPARATION DES ECHANTILLONS

masse théorique 694,4  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	692,60	686,30	695,70	689,20	687,60	691,20
quantité de principe actif	159,59	158,13	160,30	158,80	158,43	159,26

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

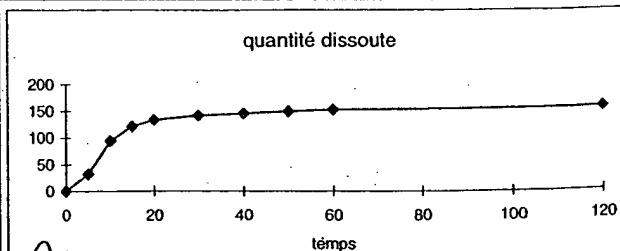
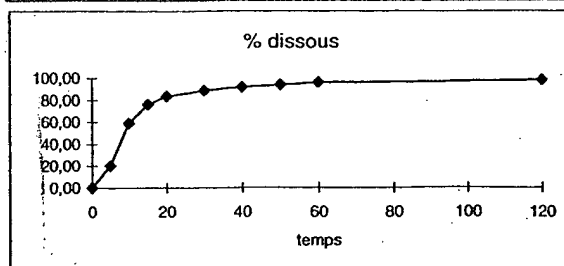
volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
5	1000	0,284	0,297	0,279	0,291	0,294	0,296
10	1000	0,872	0,859	0,841	0,868	0,819	0,812
15	1000	1,102	1,094	1,097	1,061	1,077	1,089
20	1000	1,191	1,183	1,209	1,182	1,178	1,189
30	1000	1,255	1,25	1,282	1,249	1,26	1,248
40	1000	1,295	1,292	1,285	1,297	1,298	1,279
50	1000	1,316	1,32	1,319	1,312	1,323	1,322
60	1000	1,328	1,334	1,343	1,334	1,35	1,347
120	1000	1,361	1,36	1,378	1,362	1,369	1,357

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	20,27	19,77	20,87	19,34	20,36	20,62	20,65	0,59	2,92
10,0	59,13	60,81	60,46	58,39	60,83	57,54	56,75	1,80	3,05
15,0	76,29	77,13	77,28	76,43	74,64	75,92	76,36	0,95	1,25
20,0	83,80	83,71	83,91	84,57	83,48	83,38	83,72	0,42	0,51
30,0	89,01	88,58	89,04	90,05	88,58	89,55	88,25	0,68	0,76
40,0	91,80	91,80	92,43	90,70	92,38	92,65	90,85	0,84	0,92
50,0	94,18	93,71	94,85	93,50	93,88	94,86	94,29	0,58	0,62
60,0	96,09	95,01	96,30	95,62	95,88	97,22	96,50	0,76	0,79
120,0	98,31	97,77	98,59	98,52	98,30	99,02	97,67	0,52	0,53
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	32,24	31,56	33,00	31,00	32,33	32,67	32,89	0,80	2,48
10,0	94,07	97,05	95,61	93,60	96,61	91,16	90,39	2,82	3,00
15,0	121,37	123,09	122,20	122,51	118,53	120,29	121,62	1,69	1,39
20,0	133,31	133,59	132,69	135,57	132,57	132,11	133,33	1,23	0,92
30,0	141,60	141,36	140,80	144,35	140,67	141,87	140,55	1,44	1,01
40,0	146,04	146,50	146,16	145,39	146,70	146,79	144,69	0,83	0,57
50,0	149,83	149,56	149,99	149,89	149,08	150,29	150,17	0,45	0,30
60,0	152,86	151,62	152,28	153,28	152,26	154,03	153,69	0,95	0,62
120,0	156,40	156,02	155,91	157,92	156,11	156,89	155,55	0,87	0,55
0,0									
0,0									
0,0									



RE 03.07.97

Le 10/04/97

Dissolution comprimés de F378.202

lot 2398/01 RG durée 14 Jg.

FOURNIER 1001618

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1. Préparation du LSNc 0,025%

voir p.118.

2. Pesée du milieu de dissolution galeu.

1 litre de LSNc 0,025% = 1000,0 g.

lots	Remise à jour	Pesée	Signature
1	oui	1001,0g	10/04/97 AB-10/04/97
2	oui	1001,0g	10/04/97 AB-10/04/97
3	oui	1001,0g	10/04/97 AB-10/04/97
4	oui	1001,0g	10/04/97 AB-10/04/97
5	oui	1001,0g	10/04/97 AB-10/04/97
6	oui	1001,0g	10/04/97 AB-10/04/97

3. Pesée des comprimés gel 205.

1 comprimé de 694,4 mg. contient 160 mg de fénofibrate.

Code	2398.01					
10.04.97	13:11:36					
ID	1					
	0.0 mg					
	692.6 mg					
ID						
	0.0 mg					
	693.5 mg					
ID						
	0.1 mg					
	695.9 mg					
ID						
	0.0 mg					
	691.9 mg					
ID						
	0.0 mg					
	695.5 mg					
ID						
	0.0 mg					
	691.2 mg					

AC 09.07.9

4. Conditions opératoires

densité polaire gel des équipes de palettes rouvrantes.

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  A2 10/4/97 82 10/04/92

$\sigma = 75 \text{ MPa}$

A2 10/4/97. 82 10/04/92

5. Lecture

sur KONTRON 930 gel des.

cours de 2 mm de trage optique.

filtration sur filtres H&S et SLHAC2503 Millipore.

LF 178TER COMPRIMES LOT 2398/01RG 14KG

10-04-1996 13:18

Lambda No. Valeur\_E

290.0	1	-0.0001_1	A2 air/air
290.0	2	0.0000_1	LSNa/LSNa
290.0	3	0.0000_1	A2 LSNa/LSNa
290.0	4	0.0001_1	
290.0	5	0.0001_1	
290.0	6	0.0000_1	
290.0	7	0.0002_1	
290.0	8	0.0003_1	
290.0	9	0.0003_1	
290.0	10	0.5983_1	
290.0	11	0.6219_1	
290.0	12	0.6676_1	
290.0	13	0.5703_1	
290.0	14	0.6147_1	
290.0	15	0.7075_1	
290.0	16	1.0365_1	
290.0	17	1.0580_1	
290.0	18	1.0564_1	
290.0	19	1.0471_1	
290.0	20	1.0558_1	
290.0	21	1.0704_1	
290.0	22	1.1951_1	
290.0	23	1.1971_1	
290.0	24	1.1961_1	
290.0	25	1.2136_1	
290.0	26	1.2068_1	
290.0	27	1.1906_1	
290.0	28	1.2693_1	
290.0	29	1.2779_1	
290.0	30	1.2803_1	
290.0	31	1.2821_1	
290.0	32	1.2737_1	
290.0	33	1.2456_1	
290.0	34	1.3439_1	
290.0	35	1.3287_1	
290.0	36	1.3405_1	
290.0	37	1.3615_1	
290.0	38	1.3557_1	
290.0	39	1.3064_1	
290.0	40	1.3717_1	
290.0	41	1.3737_1	
290.0	42	1.3798_1	
290.0	43	1.3928_1	
290.0	44	1.3786_1	
290.0	45	1.3389_1	
290.0	46	1.3885_1	
290.0	47	1.3931_1	
290.0	48	1.3864_1	
290.0	49	1.4103_1	
290.0	50	1.3938_1	
290.0	51	1.3528_1	
290.0	52	1.3906_1	
290.0	53	1.3858_1	
290.0	54	1.3916_1	
290.0	55	1.4096_1	
290.0	56	1.3980_1	
290.0	57	1.3567_1	

FOURNIER 1001619

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REC099K



# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
 DATE : 10/04/97  
 APPAREIL : GAL 103 GAL 108  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm: 2

TITRE : LF 178 ter 2398/01RG à 14 KG  
 N° CAHIER : 178 TER n°1 p 121  
 FICHER : m:\commun\glnq\donnbase\LF178ter\dissolution\lot 2398RG01 14 kg  
 ELUANT : LSNa 0,025 M  
 AGITATION : 75 TPM

FOURNIER 1001620

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## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	692,60	693,50	695,90	691,90	695,50	691,20
quantité de principe actif	159,59	159,79	160,35	159,42	160,25	159,26

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

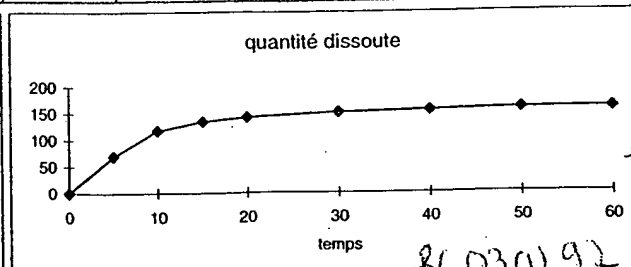
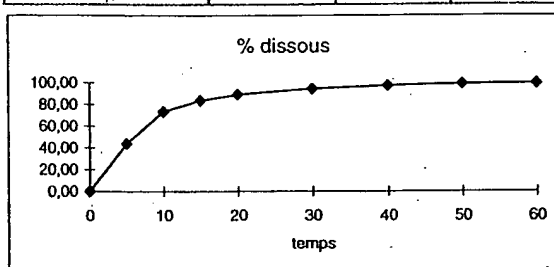
SUIVI DE LA DISSOLUTION		volume prélevé en ml 5					
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,598 ✓	0,622 ✓	0,668 ✓	0,57 ✓	0,615 ✓	0,708 ✓
10	1000	1,037 ✓	1,058 ✓	1,056 ✓	1,047 ✓	1,056 ✓	1,07 ✓
15	1000	1,195 ✓	1,197 ✓	1,196 ✓	1,214 ✓	1,207 ✓	1,191 ✓
20	1000	1,269 ✓	1,278 ✓	1,28 ✓	1,282 ✓	1,274 ✓	1,246 ✓
30	1000	1,344 ✓	1,329 ✓	1,341 ✓	1,362 ✓	1,356 ✓	1,306 ✓
40	1000	1,372 ✓	1,374 ✓	1,38 ✓	1,393 ✓	1,379 ✓	1,339 ✓
50	1000	1,389 ✓	1,393 ✓	1,386 ✓	1,41 ✓	1,394 ✓	1,353 ✓
60	1000	1,391 ✓	1,386 ✓	1,392 ✓	1,41 ✓	1,398 ✓	1,357 ✓

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	43,82	41,64	43,25	46,29	39,73	42,64	49,39	3,48	7,93
10,0	73,52	72,41	73,78	73,41	73,17	73,43	74,90	0,82	1,11
15,0	84,04	83,77	83,82	83,47	85,17	84,27	83,71	0,61	0,73
20,0	89,42	89,34	89,87	89,71	90,34	89,33	87,96	0,81	0,90
30,0	94,61	95,00	93,86	94,38	96,36	95,46	92,58	1,32	1,39
40,0	97,38	97,42	97,45	97,55	98,99	97,52	95,34	1,17	1,20
50,0	98,88	99,08	99,25	98,44	100,66	99,04	96,79	1,26	1,28
60,0	99,46	99,70	99,24	99,34	101,16	99,80	97,54	1,17	1,17
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	70,02	66,44	69,11	74,22	63,33	68,33	78,67	5,54	7,92
10,0	117,46	115,55	117,90	117,70	116,65	117,68	119,28	1,26	1,07
15,0	134,27	133,69	133,93	133,85	135,79	135,04	133,32	0,94	0,70
20,0	142,88	142,57	143,60	143,84	144,02	143,15	140,09	1,46	1,02
30,0	151,16	151,61	149,98	151,33	153,62	152,97	147,45	2,22	1,47
40,0	155,59	155,47	155,71	156,41	157,82	156,28	151,85	2,01	1,29
50,0	157,98	158,12	158,59	157,85	160,48	158,72	154,14	2,09	1,33
60,0	158,92	159,11	158,58	159,28	161,27	159,93	155,34	1,98	1,25
0,0									
0,0									
0,0									
0,0									



BL 0302.92

124 10.ch.97

LOT RG 2338/01

curete AB hy.

1. Préparation du milieu de dissolution

Voir page AB

2. Remplissage des bts de dissolution Bal GAL M

1 Lb LSNa 0,025N → 1001,0g.

bt	remise à zero	masse LSNa 0,025	Vérifications
1	oui	1001,1 g	82 10/04/97 M 10.ch.97
2	oui	1001,0 g	82 10/04/97 M 10.ch.97
3	oui	1001,1 g	82 10/04/97 M 10.ch.97
4	oui	1002,0 g	82 10/04/97 M 10.ch.97
5	oui	1001,0 g	82 10/04/97 M 10.ch.97
6	oui	1001,0 g	82 10/04/97 M 10.ch.97

Dissolvant  
GAL 091

Conditions

T° 37°C ± 0,5 82 10/04/97 M 10.ch.97

Q 75 TPA 82 10/04/97 M 10.ch.97

3. Pesée des comprimés Bal GAL WS

10.04.97 13:17:08  
Code 2398.01-18

1

0.0 mg

696.8 mg

2

0.0 mg

690.7 mg

3

0.0 mg

696.3 mg

4

0.0 mg

694.1 mg

5

0.0 mg

691.4 mg

6

0.0 mg

695.9 mg

M

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BC 03.07.97

h. Lecture Spectro GAZ 2B's chromatique GAZ 124

125

LF 178 TER LOT RG 2398/01 18 KG

04-10-1997 15:55

			Lambda	No.	Valeur_E			
1	290.0	1	290.0	1	0.0006_1	290.0	31	1.3099_1
	290.0	2		2	0.0003_1			1.3066_1
	290.0	3		3	0.0007_1			1.2914_1
2	290.0	2	290.0	4	-0.0001_1	290.0	34	1.2975_1
	290.0	2		5	0.0009_1			1.2660_1
	290.0	3		6	0.0005_1			1.2823_1
3	290.0	3	290.0	7	0.2433_1	290.0	37	1.3347_1
	290.0	3		8	0.2756_1			1.3259_1
	290.0	3		9	0.2869_1			1.3208_1
1	290.0	1	290.0	10	0.2726_1	290.0	40	1.3161_1
	290.0	1		11	0.3272_1			1.2919_1
	290.0	1		12	0.2953_1			1.3177_1
2	290.0	2	290.0	13	0.7686_1	290.0	43	1.3640_1
	290.0	2		14	0.8517_1			1.3624_1
	290.0	2		15	0.8384_1			1.3618_1
3	290.0	3	290.0	16	0.8384_1	290.0	46	1.3618_1
	290.0	3		17	0.8384_1			1.3618_1
	290.0	3		18	0.8384_1			1.3618_1

1 AZ Air / Air

2 LSNa / LSNa

3 AZ LSNa / LSNa

125

FOURNIER 1001622

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h. Lecture Spectro GAL 233

chronométré GAL 124

125

LF 178 TER LOT RG 2398/01 18 KG

04-10-1997 15:55

Lambda	No.	Valeur_E	Lambda	No.	Valeur_E
290.0	1	-0.0000_1	290.0	31	1.3099_1
290.0	2	0.0029_1	290.0	32	1.3066_1
290.0	3	-0.0001_1	290.0	33	1.2914_1
290.0	4	0.0009_1	290.0	34	1.2975_1
290.0	5	0.0005_1	290.0	35	1.2660_1
290.0	6	0.0005_1	290.0	36	1.2823_1
290.0	7	0.2433_1	290.0	37	1.3347_1
290.0	8	0.2756_1	290.0	38	1.3259_1
290.0	9	0.2869_1	290.0	39	1.3208_1
290.0	10	0.2726_1	290.0	40	1.3161_1
290.0	11	0.3272_1	290.0	41	1.2919_1
290.0	12	0.2953_1	290.0	42	1.3177_1
290.0	13	0.7686_1	290.0	43	1.3640_1
290.0	14	0.8517_1	290.0	44	1.3624_1
290.0	15	0.8384_1	290.0	45	1.3618_1
290.0	16	0.8150_1	290.0	46	1.3394_1
290.0	17	0.8940_1	290.0	47	1.3391_1
290.0	18	0.8533_1	290.0	48	1.3371_1
290.0	19	1.0974_1	290.0	49	1.3709_1
290.0	20	1.1053_1	290.0	50	1.3656_1
290.0	21	1.1121_1	290.0	51	1.3653_1
290.0	22	1.1001_1	290.0	52	1.3512_1
290.0	23	1.1118_1	290.0	53	1.3473_1
290.0	24	1.0916_1	290.0	54	1.3363_1
290.0	25	1.2065_1	290.0	55	1.4028_1
290.0	26	1.2210_1	290.0	56	1.3786_1
290.0	27	1.2098_1	290.0	57	1.3834_1
290.0	28	1.2076_1	290.0	58	1.3906_1
290.0	29	1.2100_1	290.0	59	1.3745_1
290.0	30	1.1910_1	290.0	60	1.3696_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 10/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2398/01 à 18 KG  
N° CAHIER : LF 178 TER n°1 p 125  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2398RG01 18 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

FOURNIER 1001623

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## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	696,80	690,70	696,30	694,10	691,40	695,90
quantité de principe actif	160,55	159,15	160,44	159,93	159,31	160,35

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,243	0,276	0,287	0,273	0,327	0,295
10	1000	0,769	0,852	0,838	0,815	0,894	0,853
15	1000	1,097	1,105	1,112	1,1	1,112	1,092
20	1000	1,207	1,221	1,21	1,208	1,21	1,191
30	1000	1,31	1,307	1,291	1,298	1,266	1,282
40	1000	1,335	1,326	1,321	1,316	1,292	1,318
50	1000	1,364	1,362	1,362	1,339	1,339	1,337
60	1000	1,371	1,366	1,365	1,351	1,347	1,336

PC 03.07.97

290.0 m 30 1.1910\_1 290.0 59 3745\_1  
290.0 60 1.3696\_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 10/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2  
TITRE : LF 178 TER RG 2398/01 à 18 KG  
N° CAHIER : LF-178 TER n°1 p 125  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2398RG01 18 kg  
ELUANT : LNa 0,025 M  
AGITATION : 75 TPM

Fournier 1001624

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Subject to  
Protective Order

### PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	696,80	690,70	696,30	694,10	691,40	695,90
quantité de principe actif	160,55	159,15	160,44	159,93	159,31	160,35

Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

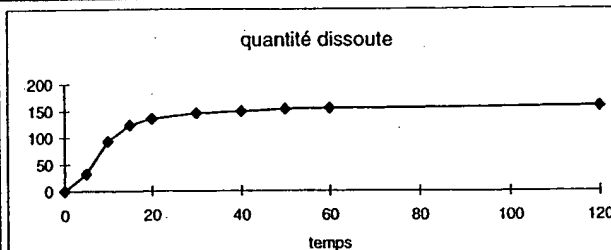
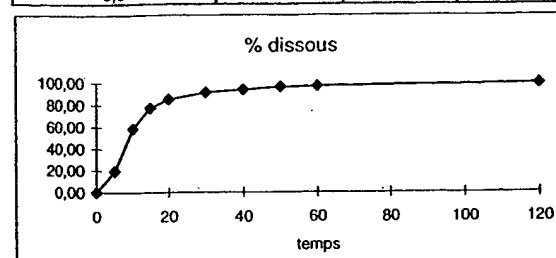
volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
5	1000	0,243	0,276	0,287	0,273	0,327	0,295
10	1000	0,769	0,852	0,838	0,815	0,894	0,853
15	1000	1,097	1,105	1,112	1,1	1,112	1,092
20	1000	1,207	1,221	1,21	1,208	1,21	1,191
30	1000	1,31	1,307	1,291	1,298	1,266	1,282
40	1000	1,335	1,326	1,321	1,316	1,292	1,318
50	1000	1,364	1,362	1,362	1,339	1,339	1,337
60	1000	1,371	1,366	1,365	1,351	1,347	1,336
120	1000	1,403	1,379	1,383	1,391	1,375	1,37

### RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	19,70	16,82	19,27	19,88	18,97	22,81	20,44	1,96	9,97
10,0	58,24	53,30	59,58	58,14	56,72	62,47	59,21	3,08	5,28
15,0	77,01	76,27	77,54	77,40	76,80	77,98	76,07	0,76	0,98
20,0	84,68	84,26	86,03	84,57	84,69	85,21	83,31	0,91	1,08
30,0	90,96	91,81	92,46	90,60	91,36	89,53	90,02	1,11	1,22
40,0	93,19	93,99	94,24	93,13	93,06	91,79	92,96	0,87	0,93
50,0	95,91	96,46	97,21	96,42	95,11	95,52	94,74	0,94	0,98
60,0	96,76	97,42	97,97	97,10	96,41	96,54	95,13	0,98	1,02
120,0	99,14	100,10	99,35	98,82	99,66	98,96	97,95	0,75	0,75
0,0									
0,0									
0,0									

### RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	31,50	27,00	30,67	31,89	30,33	36,33	32,78	3,08	9,78
10,0	93,14	85,58	94,82	93,27	90,71	99,52	94,94	4,69	5,03
15,0	123,18	122,45	123,40	124,18	122,83	124,23	121,97	0,93	0,75
20,0	135,44	135,28	136,91	135,69	135,44	135,74	133,58	1,08	0,79
30,0	145,50	147,40	147,14	145,36	146,11	142,64	144,35	1,80	1,24
40,0	149,07	150,90	149,98	149,41	148,83	146,23	149,06	1,58	1,06
50,0	153,41	154,87	154,72	154,70	152,12	152,17	151,91	1,48	0,97
60,0	154,77	156,40	155,92	155,79	154,19	153,80	152,54	1,50	0,97
120,0	158,58	160,72	158,12	158,55	159,39	157,66	157,06	1,31	0,83
0,0									
0,0									
0,0									



le 10/4/97

Dosage du fenofibrate dans les  
mélanges avant compression

lot 2397/01 RG.

lot 2398/01 RG.

dosage effectué selon procédure ALDA 004-06/05.

conditions chromatographiques voir p 99.

1. Préparation des solutions témoins (à préparer en double).

\* solutions témoins mères balance nos DAP.

introduire 25,00 mg enillon exactement connu de fenofibrate  
au 1241 dans fiole de 50 ml.

ajouter 5 ml de THF.

qsp avec ACN.

Mettre 30 ml sous agitation magnétique.

\*\*\* 10/04/97 - 10:26 \*\*\*

Echantillon: FENOFIBRATE  
No controle: ARR 1241 T1  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 7.25194 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 7.27727 g

POIDS NET : 0.02533 g

lot TH1

lot TH2

\*\*\* 10/04/97 - 10:29 \*\*\*

Echantillon: FENOFIBRATE  
No controle: ARR 1241 T2  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 11.07059 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 11.09549 g

POIDS NET : 0.02490 g

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\* solutions témoin de travail.

à l'aide d'une pipette jaugée introduire 2 ml de solution témoin mère dans fiole de 20 ml.

qsp avec phase mobile puis homogénéiser.

soit  $\gamma 1$  à  $0,566 \mu\text{g}/10 \mu\text{l}$

$\gamma 2$  à  $0,498 \mu\text{g}/10 \mu\text{l}$

2. Préparation des solutions essais (balance nos DAP).

2.1. Solution essais mères

Peser exactement environ 350 mg de mélange dans fiole de 150 ml.

\*\*\* 10/04/97 - 08:33 \*\*\*

Echantillon: LF 178TER  
No controle: 2397RG01 1  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 14.24833 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 14.60254 g

POIDS NET : 0.35421 g

\*\*\* 10/04/97 - 08:37 \*\*\*

Echantillon: LF 178TER  
No controle: 2397RG01 2  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 15.38274 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 15.73109 g

POIDS NET : 0.34835 g

\*\*\* 10/04/97 - 08:39 \*\*\*

Echantillon: LF 178TER  
No controle: 2398RG01 3  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 11.96462 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 12.31560 g

POIDS NET : 0.35098 g

\*\*\* 10/04/97 - 08:42 \*\*\*

Echantillon: LF178 TER  
No controle: 2398RG01 4  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 13.15772 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 13.50633 g

POIDS NET : 0.34861 g

ajouter 15 ml de THF puis 7,5 ml de Phobis.

mettre 45 min sous agitation magnétique.

qsp avec ACN.

mettre 45 min sous agitation magnétique.

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## 2.2. solution essai de travail

à l'aide d'une pipette jaugée introduire 1 ml de solution témoin  
mêlé dans fiole de 25 ml.

qsp avec P mobile.

homogénéiser.

## 3. Injections et résultats.

les chromatogrammes sont dans l'annexe 2.

\* calcul du facteur de calibration de T1 et le coefficient de variation.

aire	$F_{T1} = \frac{\text{conc en-fiole}}{\text{aire}}$	
343844,	$1,473 \times 10^{-6}$	$E_c = 3,5 \times 10^{-3}$ $\omega = 0,2\%$ $\bar{x} = 1,471 \times 10^{-6}$ Moyenne des 3 derniers essais $F_{T1} = 1,469 \times 10^{-6}$
343308,	$1,476 \times 10^{-6}$	
344710,	$1,470 \times 10^{-6}$	
345345,	$1,467 \times 10^{-6}$	
344761,	$1,469 \times 10^{-6}$	

\* calcul du facteur de calibration de T2.

aire	$F_{T2}$	
340021,	$1,465 \times 10^{-6}$	$F_{T2} = 1,468 \times 10^{-6}$
339166,	$1,468 \times 10^{-6}$	
338537,	$1,471 \times 10^{-6}$	

\* vérification des témoins

$$\frac{F_{T2}}{F_{T1}} \times 100 = \frac{1,468 \times 10^{-6} \times 100}{1,469 \times 10^{-6}} = 99,9\% \text{ témoins corrects acceptés.}$$

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\* facteur de calibration moyenne

$$MF = \frac{F_{Ti} + F_{Tc}}{2} = \frac{(1,468 + 1,469) \times 10^{-6}}{2} = 1,469 \times 10^{-6}$$

\* dosage du ferofolinate par compresse

voir procédure pour formule édatée

$$375 \times \frac{MF \times A_{\text{féro}} \times 694,4}{P_{\text{émai}}}$$

solution 2397/01 RG 1.

$$375 \times \frac{1,469 \times 10^{-6} \times 148210 \times 694,4}{354,21} = 160,06 \text{ mg}$$

solution 2397/01 RG 2.

$$375 \times \frac{1,469 \times 10^{-6} \times 138848 \times 694,4}{348,35} = 152,47 \text{ mg}$$

lot 2397/01 RG à 156 mg de féro / comprimé

solution 2398/01 RG 3

$$375 \times \frac{1,469 \times 10^{-6} \times 143773 \times 694,4}{350,38} = 156,70 \text{ mg}$$

solution 2398/01 RG 4.

$$375 \times \frac{1,469 \times 10^{-6} \times 139516 \times 694,4}{348,61} = 153,09 \text{ mg}$$

lot 2398/01 RG à 155 mg de féro / comprimé

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REC09.07.97

LOT RG 2399/01

durée 15 kg.

1. Préparation du milieu de dissolution

- pesée de l'eau Bal GAL 014

Tare = 2,730 kg M.M.ch.97 ~~8~~ 11/04/97

Brut = 25,630 kg M.M.ch.97 ~~8~~ 11/04/97

Net = 25,630 - 2,730 = 22,900 kg

- pesée du LSN Bal GAL 011

Tare = 153,5 g. <sup>remise à zero</sup> plus R.A.2. M.M.ch.97 CC 11/04/97

Net = 165,1 g. M.M.ch.97 ~~8~~ 11/04/97

Quantité à peser pour un milieu à 0,025N (PN = 288,4 g)

masse LSN = 22,9

11.04.97

09:45:32

Code

2399.01-15

= 165,1 g. ~~8~~ 11/04/97

2. Remplissage des bols

1 litre LSN 0

ID	remise à zero
1	oui
2	oui
3	oui
4	oui
5	oui
6	oui

ID

1

0.0 mg

689.6 mg

ID

2

0.0 mg

694.2 mg

ID

3

0.0 mg

687.5 mg

ID

4

0.0 mg

694.0 mg

ID

5

0.0 mg

690.1 mg

ID

6

0.0 mg

695.3 mg

1. GAL M

P.

1. Vérification

CC 11/04/97 M.M.ch.97

CC 11/04/97 M.M.ch.97

CC 11/04/97 M.M.ch.97

CC 11/04/97 M.M.ch.97

CC 11/04/97 M.M.ch.97

CC 11/04/97 M.M.ch.97

Dissolution

GAL 091

Cc

T° ?

0 ?

11/04/97

ch.97 ~~8~~ 37°C ± 0,

14.97 ~~8~~ 11/04/97

3. Pesée des comprimés

Bal GAL 205

FOURNIER 1001629

Highly Confidential

Subject to

Protective Order

RC 09 07.97

LOT RG 2399/01

durée 15 kg.

1. Préparation du milieu de dissolution

- pesée de l'eau Bal GAL 014

Tare = 2,730 kg M.M.ch.97 ~~CC~~ 11/04/97

Brut = 25,630 kg M.M.ch.97 ~~CC~~ 11/04/97.

Net = 25,630 - 2,730 = 22,900 kg

- pesée du LSA Bal GAL 011

Tare = 153,5 g. <sup>remise à zéro</sup> puis R.A.Z. M.M.ch.97 CC 11/04/97

Net = 165,1 g. M.M.ch.97 ~~CC~~ 11/04/97

→ Quantité à peser pour un milieu à 0,025N (PN = 288,4 g)

masse LSA = 22,900 × 288,4 × 0,025 = 165,1 g. ~~CC~~ 11/04/97.

2. Remplissage des bûles de dissolution Bal. GAL 11

1 LSA 0,025N → 1001,0 g.

bal	remise à zéro	masse LSA 0,025N	Vérificateur
1	oui	1001,0 g	CC 11/04/97 M.M.ch.97
2	oui	1001,0 g	CC 11/04/97 M.M.ch.97
3	oui	1001,0 g	CC 11/04/97 M.M.ch.97
4	oui	1001,0 g	CC 11/04/97 M.M.ch.97
5	oui	1001,0 g	CC 11/04/97 M.M.ch.97
6	oui	1001,0 g	CC 11/04/97 M.M.ch.97

Conditions

Dissolvabilité

GAL 081

T° 37°C ± 0,5 M.M.ch.97 ~~CC~~ 11/04/97

75 TPN M.M.ch.97 ~~CC~~ 11/04/97.

11/04/97

3. Pesée des comprimés

Bal GAL 205

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✓

RC 09.07.97

h. lecture

chronométrique GAI 12h

LF 178 TER LOT RG 2399/01 15 KG

04-11-1997 12:32

Lambda No. Valeur\_E

M

1 290.0 1 0.0000\_1  
2 290.0 2 0.0032\_1  
3 290.0 M 3 -0.0001\_1

1 A2 Air/Air

2 LSNe/LSNe

3 A2 LSNe/LSNe

290.0  $\tau_0$  1 -0.0029\_1  
290.0 2 -0.0001\_1  
290.0 3 0.0002\_1  
290.0 4 0.0000\_1  
290.0 5 0.0001\_1  
290.0 6 -0.0003\_1  
290.0  $\tau_5$  7 0.3166\_1  
290.0 8 0.3250\_1  
290.0 9 0.3696\_1  
290.0 10 0.3568\_1  
290.0 11 0.3505\_1  
290.0 12 0.3429\_1  
290.0  $\tau_{10}$  13 0.8736\_1  
290.0 14 0.8900\_1  
290.0 15 0.9225\_1

290.0  $\tau_{30}$  31 1.2847\_1  
290.0 32 1.3058\_1  
290.0 33 1.2842\_1  
290.0 34 1.2638\_1  
290.0 35 1.2707\_1  
290.0 36 1.2911\_1  
290.0  $\tau_{40}$  37 1.3262\_1  
290.0 38 1.3467\_1  
290.0 39 1.3021\_1  
290.0 40 1.3239\_1  
290.0 41 1.3121\_1  
290.0 42 1.3227\_1  
290.0  $\tau_{50}$  43 1.3396\_1  
290.0 44 1.3658\_1  
290.0 45 1.3197\_1  
290.0 46 1.3260\_1

FOURNIER 1001631  
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Subject to  
Protective Order

110 5007.007

h. lecture

chronométré GAL 12h

LF 178 TER LOT RG 2399/01 15 KG

04-11-1997 12:32

Lambda No. Valeur\_E

1 290.0 1 0.0000\_1  
2 290.0 2 0.0032\_1  
3 290.0 3 -0.0001\_1

1 A2 Air/A2  
2 LSNu/LSNu  
3 A2 LSNu/LSNu

290.0	1	-0.0029_1
290.0	2	-0.0001_1
290.0	3	0.0002_1
290.0	4	0.0000_1
290.0	5	0.0001_1
290.0	6	-0.0003_1
290.0	7	0.3166_1
290.0	8	0.3250_1
290.0	9	0.3696_1
290.0	10	0.3568_1
290.0	11	0.3505_1
290.0	12	0.3429_1
290.0	13	0.8736_1
290.0	14	0.8900_1
290.0	15	0.9225_1
290.0	16	0.9137_1
290.0	17	0.8835_1
290.0	18	0.8851_1
290.0	19	1.1046_1
290.0	20	1.1173_1
290.0	21	1.1046_1
290.0	22	1.1072_1
290.0	23	1.1044_1
290.0	24	1.1077_1
290.0	25	1.2043_1
290.0	26	1.2179_1
290.0	27	1.1905_1
290.0	28	1.2003_1
290.0	29	1.1974_1
290.0	30	1.2059_1

290.0	31	1.2847_1
290.0	32	1.3058_1
290.0	33	1.2842_1
290.0	34	1.2638_1
290.0	35	1.2707_1
290.0	36	1.2911_1
290.0	37	1.3262_1
290.0	38	1.3467_1
290.0	39	1.3021_1
290.0	40	1.3239_1
290.0	41	1.3121_1
290.0	42	1.3227_1
290.0	43	1.3396_1
290.0	44	1.3658_1
290.0	45	1.3197_1
290.0	46	1.3260_1
290.0	47	1.3264_1
290.0	48	1.3436_1
290.0	49	1.3536_1
290.0	50	1.3635_1
290.0	51	1.3201_1
290.0	52	1.3363_1
290.0	53	1.3459_1
290.0	54	1.3501_1
290.0	55	1.3901_1
290.0	56	1.4039_1
290.0	57	1.3563_1
290.0	58	1.3826_1
290.0	59	1.3758_1
290.0	60	1.3827_1

DISSOLUTION

m:\commun\glnq\traitdon\distern5  
date édition: le 06/02/97

CONDITIONS ET MODES OPERATOIRES

OPERATEUR: D.LECRIT  
DATE: 11/04/97  
APPAREIL: GAL 233 GAL 091  
LONGUEUR D'ONDE: 290 nm  
CUVE en mm: 2

TITRE: LF 178 TER RG 2399/01 à 15 KG  
N° CAHIER: LF 178 TER n°1 p 131.  
FICHIER: M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2399RG01 15 kg  
ELUANT: LSNu 0,025 M  
AGITATION: 75 TPM

FOURNIER 1001632

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Subject to  
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PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	689,60	694,20	687,50	694,00	690,10	695,30
quantité de principe actif	158,89	159,95	158,41	159,91	159,01	160,21

Témoin 100mg/l 0,900

SUIVI DE LA DISSOLUTION

volume prélevé en ml		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
5	1000	0,317	0,325	0,37	0,357	0,351	0,343
10	1000	0,874	0,89	0,923	0,914	0,884	0,885
15	1000	1,105	1,117	1,105	1,107	1,104	1,108
20	1000	1,204	1,218	1,191	1,2	1,197	1,206
30	1000	1,285	1,306	1,284	1,264	1,271	1,291
40	1000	1,326	1,347	1,302	1,324	1,312	1,323
50	1000	1,34	1,366	1,32	1,326	1,326	1,344

PC 09.07.97

# DISSOLUTION

m:\commun\glnq\traiton\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 11/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2

TITRE : LF 178 TER RG 2399/01 à 15 KG  
N° CAHIER : LF 178 TER n°1 p 131  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2399RG01 15 kg  
ELUANT : LNa 0,025 M  
AGITATION : 75 TPM

Fournier 1001633

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Protective Order

## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	689,60	694,20	687,50	694,00	690,10	695,30
quantité de principe actif	158,89	159,95	158,41	159,91	159,01	160,21

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

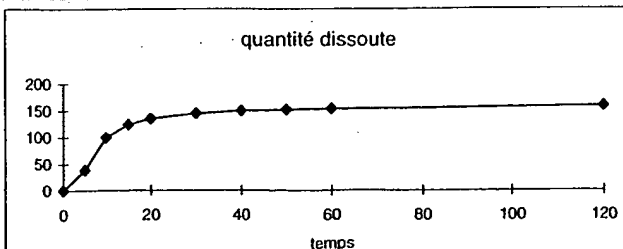
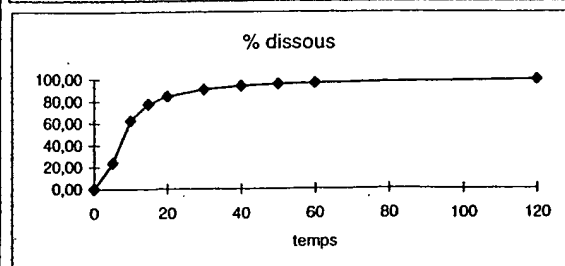
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,317	0,325	0,37	0,357	0,351	0,343
10	1000	0,874	0,89	0,923	0,914	0,884	0,885
15	1000	1,105	1,117	1,105	1,107	1,104	1,108
20	1000	1,204	1,218	1,191	1,2	1,197	1,206
30	1000	1,285	1,306	1,284	1,264	1,271	1,291
40	1000	1,326	1,347	1,302	1,324	1,312	1,323
50	1000	1,34	1,366	1,32	1,326	1,326	1,344
60	1000	1,354	1,364	1,32	1,336	1,346	1,35
120	1000	1,39	1,404	1,356	1,383	1,376	1,383

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	23,97	22,17	22,58	25,95	24,81	24,53	23,79	1,43	5,95
10,0	62,51	61,23	61,94	64,87	63,63	61,89	61,50	1,43	2,29
15,0	77,64	77,69	78,01	77,96	77,36	77,58	77,27	0,30	0,39
20,0	84,65	85,00	85,42	84,38	84,21	84,46	84,45	0,46	0,54
30,0	90,71	91,08	91,95	91,32	89,07	90,05	90,76	1,02	1,12
40,0	93,86	94,40	95,26	93,03	93,68	93,36	93,43	0,82	0,88
50,0	95,34	95,84	97,04	94,75	94,28	94,79	95,35	0,99	1,04
60,0	96,37	97,29	97,38	95,22	95,43	96,66	96,23	0,91	0,95
120,0	99,41	100,28	100,63	98,20	99,16	99,22	98,99	0,89	0,90
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	38,20	35,22	36,11	41,11	39,67	39,00	38,11	2,21	5,79
10,0	99,64	97,29	99,07	102,76	101,75	98,42	98,52	2,14	2,14
15,0	123,76	123,44	124,79	123,50	123,71	123,35	123,79	0,53	0,43
20,0	134,93	135,05	136,63	133,67	134,65	134,30	135,30	1,01	0,75
30,0	144,58	144,72	147,08	144,66	142,43	143,19	145,41	1,65	1,14
40,0	149,61	149,99	152,36	147,37	149,80	148,45	149,69	1,68	1,12
50,0	151,98	152,28	155,22	150,10	150,76	150,73	152,75	1,89	1,24
60,0	153,61	154,58	155,76	150,83	152,61	153,69	154,17	1,71	1,11
120,0	158,47	159,34	160,96	155,56	158,57	157,77	158,58	1,78	1,13
0,0									
0,0									
0,0									



le 11/4/97

Dosage du fénofibrate contenu  
dans les comprimés de FJ73 ser.  
mélange lot 2399/01 RG.

dosage effectué selon procédure ALDAP004.06/05

conditions chromatographiques. voir p.99.

1. Préparation des solutions témoins (à préparer en double).

\* solutions témoins nées balance nos DAP

introduire 25,00 mg de fénofibrate Au #1241 enflon exactement  
comme dans fiole de 50 ml.

ajouter 5 ml de THF.

qsp avec ACN.

Mettre 30 min sous agitation magnétique.

\*\*\* 11/04/97 - 08:46 \*\*\*

Echantillon: FENOFIBRATE  
No controle: ARR 1241 T1  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 11.51415 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 11.53883 g

POIDS NET : 0.02468 g

\*\*\* 11/04/97 - 08:48 \*\*\*

Echantillon: FENOFIBRATE  
No controle: ARR 1241 T2  
Operateur : AG

\_\_\_\_\_ 0 \_\_\_\_\_

Poids tare : 10.87260 g

\_\_\_\_\_ 0 \_\_\_\_\_

Poids brut : 10.89800 g

POIDS NET : 0.02540 g

voir T1 et T2.

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\* numéros sérologiques de suivi

Le 11/04/97

à l'aide d'une pipette jaugée, introduire 2 ml de solution  
témoin n°1 dans fiole de 20 ml.

qsp avec phase mobile.

homogénéiser.

noir 1 à 0,4936 µg / 10 µl.

noir 2 à 0,5080 µg / 10 µl.

## 2. Préparation des solutions essais

balance nos DAR.

### 2.1. solutions essais mères

Peser exactement environ 350,00 mg de mélange dans fiole de 150 ml.

\*\*\* 11/04/97 - 08:30 \*\*\*

Echantillon: LF 178TER  
No controle: LOT 2399RG01

1  
Opérateur : AG

0

Poids tare : 18.88493 g

0

Poids brut : 19.23615 g

POIDS NET : 0.35122 g

\*\*\* 11/04/97 - 08:33 \*\*\*

Echantillon: LF 178TER  
No controle: LOT 2399RG01

2  
Opérateur : AG

0

Poids tare : 17.79002 g

0

Poids brut : 18.14293 g

POIDS NET : 0.35291 g

ajouter 15 ml de THF + 7,5 ml de φ mobile.

Mettre 45 min sous agitation magnétique.

qsp avec ACN.

Mettre 45 min sous agitation magnétique.

### 2.2. Solutions essais de travail

à l'aide d'une pipette jaugée introduire 1 ml de solution témoin  
n°1 dans fiole de 25 ml.

qsp avec φ mobile.

homogénéiser.

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PC 09.07.97



## 3 Injection et résultats.

Les chromatogrammes sont de l'annexe 2.

\* calcul du coef de variation et du fac de calibration de T<sub>1</sub>.

aire	$F_{T_1} = \frac{\text{conc en fase de } T_1}{\text{aire}}$
335169.	$1,473 \times 10^{-6}$
333526/	$1,480 \times 10^{-6}$
335484/	$1,471 \times 10^{-6}$
333758,	$1,479 \times 10^{-6}$
333111./	$1,482 \times 10^{-6}$

$$E_c = 4,74 \times 10^{-2}$$

$$\omega = 0,3\%$$

$$\bar{x} = 1,477 \times 10^{-6}$$

$$F_{T_1} = 1,477 \times 10^{-6}$$

\* facteur de calibration de T<sub>2</sub>.

aire	$F_{T_2}$
343553/	$1,479 \times 10^{-6}$
343524/	$1,479 \times 10^{-6}$
342710./	$1,482 \times 10^{-6}$

$$F_{T_2} = 1,480 \times 10^{-6}$$

\* vérifications des témoins

$$\frac{F_{T_2}}{F_{T_1}} \times 100 = \frac{1,480 \times 10^{-6}}{1,477 \times 10^{-6}} = 100,2\% \quad \text{témoins corrects après}$$

\* facteur de calibration moyen

$$PF = \frac{F_{T_2} + F_{T_1}}{2} = \frac{1,480 + 1,477}{2} = 1,479 \times 10^{-6}$$

\* dosage du fin film / comprimé

voir procédure pour formule séparée.

$$375 \times \frac{PF \times \text{aire essai} \times 694,9}{P_{\text{essai}}}$$

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RC 09 07 97

$$375 \times \frac{1,479 \times 10^{-6} \times 148214 \times 6944}{351,22} = 162,52 \text{ mg.}$$

solution- 2399/01 2.

$$375 \times \frac{1,479 \times 10^{-6} \times 149823 \times 6944}{0,5 \times 352,91} = 163,50 \text{ mg.}$$

lot 2399/01 RG à 163 mg / comprimé.

le 11/4/97

Démolition comprimé F178 Jca  
lot 2399/01 RG  
densité 20 kg.

1. Préparation du LSA 0,025 H

voir p130.

2. Pesée du résidu de démolition. galien.

1 l de LSA 0,025 H = 1001,0 g.

hop	Remise à gis	Pesée	Signature.
1	oui	1001,0 g.	R 11/04/97 R 11/04/97
2	oui	1001,0 g	R 11/04/97 R 11/04/97
3	oui	1001,0 g	R 11/04/97 R 11/04/97
4	oui	1001,0 g.	R 11/04/97 R 11/4/97
5	oui	1001,0 g.	R 11/04/97 R 11/4/97
6	oui	1001,0 g.	R 11/04/97 R 11/4/97.

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RC09.07.97

conditions

$T = 37^{\circ}\text{C} \pm 0.5$  M Moh. 92 de 21/1/57

② 75 TPN M.M.H. 97 Ae 11/4/97.

3. Prescription des comprimés Bal GAT 205

11.04.97	13:00:25
Code	2399.01-20

M

101

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695.0 mg

21 ID 2

Bill D. G.

ॐ नमो भगवते वासुदेवाय

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5. 3. 2.

5. 8. 2013



Dr. B. B.

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2. 2. 2.

2011 05.26.09

## h. lecture

chronométrie GAL 124

LF 178 TER LOT RG 2399/01 20 KG

11-04-1976 14:41

12

Lambda	No.	Valeur_E
290.0	1	0.0001_1
290.0	2	0.0016_1
290.0	3	0.0001_1

M

1. AZ Air/Air

 $2 \text{ LiNa} / \text{LiNa}$ 

3 AZ LSNu/LSNu

Lambda	No.	Valeur_E
290.0	1	-0.0001_1
290.0	2	0.0001_1
290.0	3	-0.0007_1
290.0	4	-0.0006_1
290.0	5	-0.0008_1
290.0	6	-0.0007_1
290.0	7	0.2173_1
290.0	8	0.2154_1
290.0	9	0.2435_1
290.0	10	0.2332_1
290.0	11	0.2798_1
290.0	12	0.2225_1
290.0	13	0.7768_1
290.0	14	0.8035_1
290.0	15	0.7789_1
290.0	16	0.7770_1
290.0	17	0.8438_1
290.0	18	0.7800_1
290.0	19	1.0807_1
290.0	20	1.0808_1
290.0	21	1.0908_1
290.0	22	1.0653_1
290.0	23	1.0745_1
290.0	24	1.0701_1
290.0	25	1.2063_1
290.0	26	1.1921_1
290.0	27	1.2092_1
290.0	28	1.1845_1
290.0	29	1.1813_1

290.0	31	1.2751	1
290.0	$\tau_{30}'$ 32	1.2713	1
290.0	33	1.2948	1
290.0	34	1.2543	1
290.0	35	1.2532	1
290.0	36	1.2569	1
290.0	37	1.3217	1
290.0	$\tau_{40}'$ 38	1.3021	1
290.0	39	1.3177	1
290.0	40	1.3003	1
290.0	41	1.2893	1
290.0	42	1.3146	1
290.0	43	1.3317	1
290.0	$\tau_{50}'$ 44	1.3246	1
290.0	45	1.3421	1
290.0	46	1.3256	1
290.0	47	1.3015	1
290.0	48	1.3429	1
290.0	49	1.3551	1
290.0	$\tau_{60}'$ 50	1.3368	1
290.0	51	1.3683	1
290.0	52	1.3454	1
290.0	53	1.3233	1
290.0	54	1.3545	1
290.0	55	1.3905	1
290.0	$\tau_{70}'$ 56	1.3843	1
290.0	57	1.4030	1
290.0	58	1.4035	1
290.0	59	1.3650	1
290.0	60	1.3947	1

M. barnard

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# DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 11/04/97  
APPAREIL : GAL 103 GAL 108  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2399/01 à 20 KG  
N° CAHIER : 178 TER n°1 p 136  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2399RG01 20 kg  
ELUANT : LNa 0,025 M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	696,00 /	693,00 /	694,90 /	693,80 /	687,80 /	693,00 /
quantité de principe actif	160,37	159,68	160,12	159,86	158,48	159,68

Témoin 100mg/l 0,900

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## SUIVI DE LA DISSOLUTION

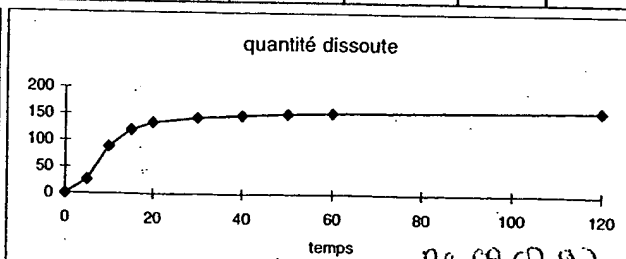
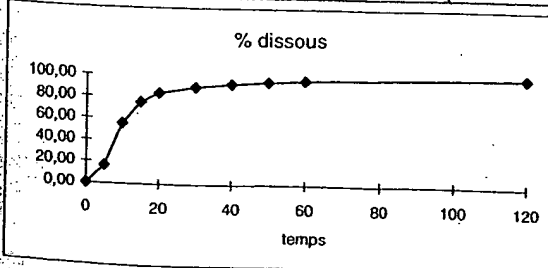
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,217 /	0,215 /	0,244 /	0,233 /	0,28 /	0,223 /
10	1000	0,777 /	0,804 /	0,779 /	0,777 /	0,844 /	0,78 /
15	1000	1,081 /	1,081 /	1,091 /	1,065 /	1,075 /	1,07 /
20	1000	1,206 /	1,192 /	1,209 /	1,185 /	1,181 /	1,18 /
30	1000	1,275 /	1,271 /	1,295 /	1,254 /	1,253 /	1,257 /
40	1000	1,322 /	1,302 /	1,318 /	1,3 /	1,289 /	1,315 /
50	1000	1,332 /	1,325 /	1,342 /	1,326 /	1,302 /	1,343 /
60	1000	1,355 /	1,337 /	1,368 /	1,345 /	1,323 /	1,355 /
120	1000	1,391 /	1,384 /	1,403 /	1,404 /	1,365 /	1,395 /

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	16,38	15,03	14,96	16,93	16,19	19,63	15,52	1,76	10,74
10,0	55,30	53,91	56,02	54,14	54,09	59,27	54,35	2,09	3,79
15,0	75,30	75,24	75,58	76,06	74,37	75,76	74,80	0,63	0,84
20,0	83,68	84,28	83,68	84,63	83,08	83,57	82,83	0,69	0,82
30,0	89,33	89,47	89,59	91,02	88,29	89,03	88,60	0,96	1,08
40,0	92,57	93,17	92,19	93,06	91,92	92,00	93,07	0,59	0,64
50,0	94,46	94,32	94,24	95,19	94,18	93,36	95,48	0,76	0,81
60,0	96,23	96,38	95,54	97,46	95,96	95,29	96,78	0,81	0,84
120,0	99,71	99,34	99,27	100,36	100,53	98,70	100,04	0,72	0,72
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	26,15	24,11	23,89	27,11	25,89	31,11	24,78	2,71	10,37
10,0	88,30	86,45	89,45	86,69	86,46	93,93	86,79	2,99	3,39
15,0	120,26	120,66	120,68	121,79	118,89	120,07	119,45	1,02	0,85
20,0	133,63	135,15	133,61	135,51	132,82	132,44	132,26	1,40	1,05
30,0	142,67	143,49	143,05	145,74	141,14	141,10	141,47	1,81	1,27
40,0	147,83	149,42	147,20	149,01	146,95	145,80	148,62	1,40	0,95
50,0	150,86	151,27	150,48	152,41	150,56	147,96	152,46	1,66	1,10
60,0	153,69	154,56	152,55	156,04	153,41	151,01	154,54	1,76	1,15
120,0	159,23	159,31	158,52	160,69	160,71	156,42	159,74	1,61	1,01
0,0									
0,0									
0,0									



me 09.07.97

138 14.04.97

Lot RG 2400/04

14 kg.

# 1. Préparation du milieu de dissolution

- pesée de l'eau Bal CAL 014

Tare = 2,740 kg. M 14.04.97 cc 14/04/97

Brut = 13,780 kg. M 14.04.97 cc 14/04/97

Net = 13,780 - 2,740 = 11,040 kg

- pesée du LSNa Bal CAL 011

pour un milieu à 0,025N =>

Masse de LSNa = 11,040 x 0,025 x 288,4 = 122,9 g.

Tare = 153,4 g. puis PAZ M 14.04.97 cc 14/04/97

Net = 122,9 g. M 14.04.97 cc 14/04/97

# 2. Remplissage des bds de dissolution Bal CAL 111

1 litre LSNa 0,025N -> 1001,0 g.

bd	remise à zero	masse LSNa 0,025N	Vérificateurs
1	oui	1001,1 g	cc 14/04/97 M 14.04.97
2	oui	1001,1 g	cc 14/04/97 M 14.04.97
3	oui	1001,0 g	cc 14/04/97 M 14.04.97
4	oui	1001,0 g	cc 14/04/97 M 14.04.97
5	oui	1001,0 g	cc 14/04/97 M 14.04.97
6	oui	1001,0 g	cc 14/04/97 M 14.04.97

conditions

T° 32°C ± 0,5 M 14.04.97 cc 14/04/97

Q 75 TPN M 14.04.97 cc 14/04/97

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# 2. Revue des compoantes Mat GAL 205

137

*M*

14.04.97 09:50:44  
Code 2400.01-14

ID

0.0 mg

695.3 mg

ID

0.0 mg

692.3 mg

ID

0.0 mg

693.3 mg

ID

0.0 mg

697.5 mg

ID

\* ou bi d'impression de la

699.2 mg

ID

0.0 mg

696.5 mg

## h. lecture

chronometre GAL 124

LF 178 TER LOT RG 2400/01 14 KG

04-14-1997 12:29

Lambda No. Valeur\_E

1 290.0 1 -0.0000\_1  
2 290.0 2 0.0026\_1  
3 290.0 3 0.0000\_1

1 AZ Air / Air

2 LSN<sub>u</sub> / LSN<sub>e</sub>

3 AZ LSN<sub>e</sub> / LSN<sub>e</sub>

290.0 T<sub>0</sub> 1 -0.0003\_1  
290.0 2 0.0003\_1  
290.0 3 0.0001\_1  
290.0 4 0.0002\_1  
290.0 5 0.0008\_1  
290.0 6 0.0007\_1  
290.0 T<sub>5</sub> 7 0.3511\_1  
290.0 8 0.3548\_1  
290.0 9 0.3747\_1  
290.0 10 0.3613\_1  
290.0 11 0.3214\_1  
290.0 12 0.3816\_1  
290.0 T<sub>10</sub> 13 0.9481\_1  
290.0 14 0.9305\_1  
290.0 15 0.9539\_1  
290.0 16 0.9587\_1  
290.0 17 0.9229\_1  
290.0 18 0.9129\_1  
290.0 T<sub>15</sub> 19 1.1540\_1  
290.0 20 1.1325\_1  
290.0 21 1.1437\_1  
290.0 22 1.1706\_1  
290.0 23 1.1485\_1  
290.0 24 1.1418\_1  
290.0 T<sub>20</sub> 25 1.2371\_1  
290.0 26 1.2383\_1  
290.0 27 1.2412\_1  
290.0 28 1.2557\_1  
290.0 29 1.2397\_1  
290.0 30 1.2300\_1

290.0 T<sub>30</sub> 31 1.3063\_1  
290.0 32 1.2940\_1  
290.0 33 1.2866\_1  
290.0 34 1.3260\_1  
290.0 35 1.3075\_1  
290.0 36 1.3038\_1  
290.0 T<sub>40</sub> 37 1.3459\_1  
290.0 38 1.3496\_1  
290.0 39 1.3395\_1  
290.0 40 1.3588\_1  
290.0 41 1.3624\_1  
290.0 42 1.3274\_1  
290.0 T<sub>50</sub> 43 1.3670\_1  
290.0 44 1.3533\_1  
290.0 45 1.3584\_1  
290.0 46 1.3668\_1  
290.0 47 1.3644\_1  
290.0 48 1.3537\_1  
290.0 T<sub>60</sub> 49 1.3711\_1  
290.0 50 1.3748\_1  
290.0 51 1.3584\_1  
290.0 52 1.3869\_1  
290.0 53 1.3974\_1  
290.0 54 1.3582\_1  
290.0 T<sub>70</sub> 55 1.3703\_1  
290.0 56 1.4095\_1  
290.0 57 1.3874\_1  
290.0 58 1.4185\_1  
290.0 59 1.4328\_1  
290.0 60 1.3728\_1

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RL 09.09.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 14/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2

TITRE : LF 178 TER RG 2400/01 à 14 KG  
N° CAHIER : LF 178 TER n°1 p 138  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2400RG01 14 kg  
ELUANT : LNa 0,025 M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	695,30	692,30	693,30	697,50	699,20	696,50
quantité de principe actif	160,21	159,52	159,75	160,71	161,11	160,48

Témoin 100mg/l 0,900

**Fournier 1001642**  
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Protective Order

## SUIVI DE LA DISSOLUTION

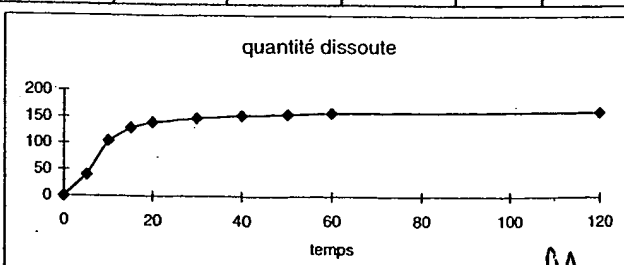
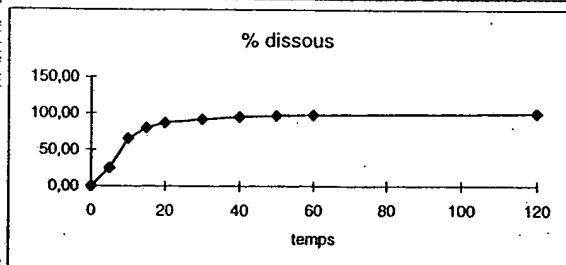
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,351	0,355	0,375	0,361	0,321	0,382
10	1000	0,948	0,931	0,954	0,959	0,923	0,913
15	1000	1,154	1,133	1,144	1,171	1,149	1,142
20	1000	1,237	1,238	1,241	1,256	1,24	1,23
30	1000	1,306	1,294	1,287	1,326	1,308	1,304
40	1000	1,346	1,35	1,34	1,359	1,362	1,327
50	1000	1,367	1,353	1,358	1,367	1,364	1,354
60	1000	1,371	1,375	1,358	1,387	1,397	1,358
120	1000	1,37	1,41	1,387	1,419	1,433	1,373

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	24,78	24,34	24,73	26,08	24,96	22,14	26,45	1,53	6,17
10,0	65,14	65,87	64,97	66,49	66,43	63,77	63,34	1,35	2,07
15,0	80,08	80,49	79,37	80,03	81,41	79,67	79,51	0,77	0,96
20,0	86,82	86,64	87,08	87,18	87,70	86,35	86,00	0,61	0,71
30,0	91,68	91,86	91,41	90,81	92,97	91,46	91,55	0,72	0,79
40,0	95,12	95,08	95,76	94,94	95,71	95,64	93,60	0,82	0,86
50,0	96,50	97,01	96,44	96,66	96,73	96,25	95,92	0,38	0,40
60,0	97,93	97,76	98,44	97,13	98,59	98,99	96,67	0,90	0,92
120,0	100,09	98,16	101,36	99,62	101,28	101,96	98,18	1,68	1,68
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	39,72	39,00	39,44	41,67	40,11	35,67	42,44	2,38	6,00
10,0	104,42	105,53	103,64	106,21	106,76	102,73	101,66	2,05	1,96
15,0	128,37	128,94	126,60	127,85	130,84	128,36	127,61	1,44	1,12
20,0	139,17	138,81	138,90	139,26	140,94	139,11	138,02	0,97	0,69
30,0	146,95	147,16	145,81	145,06	149,42	147,35	146,93	1,49	1,02
40,0	152,48	152,33	152,75	151,67	153,82	154,08	150,21	1,43	0,94
50,0	154,69	155,41	153,83	154,41	155,46	155,06	153,94	0,72	0,47
60,0	156,98	156,62	157,03	155,17	158,44	159,48	155,14	1,74	1,11
120,0	160,45	157,27	161,68	159,14	162,77	164,26	157,56	2,88	1,80
0,0									
0,0									
0,0									



RC 09.07.97

### 1. Préparation du milieu de dissolution

Var page BB

2. Remplissage des bts de dissolution Bel CIAL M

Min LGW 0025N  $\rightarrow$  100,0g.

bal	remide a zero	masse LSNa 0,025N	Verificatore.
1	oui	1001,0 g	2 14/04/82 M. Th. Ch. G.
2	oui	1001,0 g	2 14/04/82 M. Th. Ch. G.
3	oui	1001,1 g	2 14/04/82 M. Th. Ch. G.
4	oui	1001,1 g	2 14/04/82 M. Th. Ch. G.
5	oui	1001,1 g	2 14/04/82 M. Th. Ch. G.
6	oui	1001,0 g	2 14/04/82 M. Th. Ch. G.

## Conclusions

7<sup>o</sup> 37°C ± 0,5 ~~se~~ 14/04/92 Mh. Mh. oh. cl7

⑦ 75 TPN Sz 14/04/92 Mr. H. H. Gf

### 3. Revue des comprimés BulGAL205

ID	Code	14.04.97	13:37:34
ID		0.0 mg	2400.01-18
ID		688.7 mg	
ID		0.0 mg	
ID		690.0 mg	
ID		0.0 mg	
ID		690.3 mg	
ID		0.0 mg	
ID		691.8 mg	
ID		0.0 mg	
ID		694.7 mg	
ID		0.0 mg	
ID		692.4 mg	

M

**FOURNIER 1001643**

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RC 09.07.97



## h. Lechiure

Chronomètre BNL 124

LF 178 TER LOT RG 2400/01 18 KG

04-14-1997 16:04

M. Lambda No. Valeur\_E

1	290.0	1	0.0000	1
2	290.0	2	0.0027	1
3	290.0	3	0.0000	1

1. A2 Air/Air

2  
2 LSN<sub>2</sub> / LSN<sub>2</sub>

3 AZ LSN<sub>1</sub>/LSN<sub>2</sub>

290.0	1	0.0008
290.0	2	0.0000
290.0	3	0.0000
290.0	4	0.0006
290.0	5	-0.0002
290.0	6	0.0001
290.0	7	0.2140
290.0	8	0.2118
290.0	9	0.1918
290.0	10	0.2053
290.0	11	0.2012
290.0	12	0.1969
290.0	13	0.7753
290.0	14	0.7563
290.0	15	0.7608
290.0	16	0.7332

290.0	$\tau_{30}$	31	1.2791	1
290.0		32	1.2752	1
290.0		33	1.2727	1
290.0		34	1.2924	1
290.0		35	1.2519	1
290.0		36	1.2733	1
290.0	$\tau_{40}$	37	1.3161	1
290.0		38	1.3012	1
290.0		39	1.2999	1
290.0		40	1.3530	1
290.0		41	1.3285	1
290.0		42	1.3113	1
290.0	$\tau_{50}$	43	1.3393	1
290.0		44	1.3404	1
290.0		45	1.3159	1
290.0		46	1.3510	1
290.0		47	1.3530	1
290.0		48	1.3516	1

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54

h. Lecture

chronometre OAL 124

LF 178 TER LOT RG 2400/01 18 KG

M

Lambda No. Valeur\_E

04-14-1997 16:04

1 290.0 1 0.0000\_1  
2 290.0 2 0.0027\_1  
3 290.0 3 0.0000\_1

M

1 AZ Air/Air

2 LSN<sub>Na</sub>/LSN<sub>Na</sub>

3 AZ LSN<sub>Na</sub>/LSN<sub>Na</sub>

290.0 1 0.0008\_1  
290.0 2 0.0000\_1  
290.0 3 0.0000\_1  
290.0 4 0.0006\_1  
290.0 5 -0.0002\_1  
290.0 6 0.0001\_1  
290.0 7 0.2140\_1  
290.0 8 0.2118\_1  
290.0 9 0.1918\_1  
290.0 10 0.2053\_1  
290.0 11 0.2012\_1  
290.0 12 0.1969\_1  
290.0 13 0.7753\_1  
290.0 14 0.7563\_1  
290.0 15 0.7608\_1  
290.0 16 0.7332\_1  
290.0 17 0.7237\_1  
290.0 18 0.7256\_1  
290.0 19 1.0699\_1  
290.0 20 1.0701\_1  
290.0 21 1.0771\_1  
290.0 22 1.0649\_1  
290.0 23 1.0382\_1  
290.0 24 1.0533\_1  
290.0 25 1.1916\_1  
290.0 26 1.1731\_1  
290.0 27 1.1785\_1  
290.0 28 1.1975\_1  
290.0 29 1.1851\_1  
290.0 30 1.1981\_1

290.0 31 1.2791\_1  
290.0 32 1.2752\_1  
290.0 33 1.2727\_1  
290.0 34 1.2924\_1  
290.0 35 1.2519\_1  
290.0 36 1.2733\_1  
290.0 37 1.3161\_1  
290.0 38 1.3012\_1  
290.0 39 1.2999\_1  
290.0 40 1.3530\_1  
290.0 41 1.3285\_1  
290.0 42 1.3113\_1  
290.0 43 1.3393\_1  
290.0 44 1.3404\_1  
290.0 45 1.3159\_1  
290.0 46 1.3510\_1  
290.0 47 1.3530\_1  
290.0 48 1.3516\_1  
290.0 49 1.3559\_1  
290.0 50 1.3570\_1  
290.0 51 1.3438\_1  
290.0 52 1.3840\_1  
290.0 53 1.3586\_1  
290.0 54 1.3686\_1  
290.0 55 1.3750\_1  
290.0 56 1.3803\_1  
290.0 57 1.3556\_1  
290.0 58 1.4123\_1  
290.0 59 1.3898\_1  
290.0 60 1.3871\_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 14/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2400/01 à 18 KG  
N° CAHIER : LF 178 TER n°1 p 142  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2400RG01 18 kg  
ELUANT : LSN<sub>Na</sub> 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai 688,70	690,00	690,30	691,80	694,70	692,40
quantité de principe actif 158,69	158,99	159,06	159,40	160,07	159,54

FOURNIER 1001645

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Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,214	0,212	0,192	0,205	0,201	0,197
10	1000	0,775	0,756	0,761	0,733	0,724	0,726
15	1000	1,07	1,07	1,077	1,065	1,038	1,053
20	1000	1,192	1,173	1,179	1,198	1,185	1,198
30	1000	1,279	1,275	1,273	1,292	1,252	1,273
40	1000	1,316	1,301	1,3	1,353	1,329	1,311
50	1000	1,339	1,34	1,316	1,351	1,353	1,352
60	1000	1,356	1,357	1,344	1,384	1,359	1,360

PC 0302.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 14/04/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVES en mm : 2

TITRE : LF 178 TER RG 2400/01 à 18 KG  
 N° CAHIER : LF 178 TER n°1 p 142  
 FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2400RG01 18 kg  
 ELUANT : LSNa 0,025 M  
 AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	688,70 /	690,00 /	690,30 /	691,80 /	694,70 /	692,40 /
quantité de principe actif	158,69	158,99	159,06	159,40	160,07	159,54

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

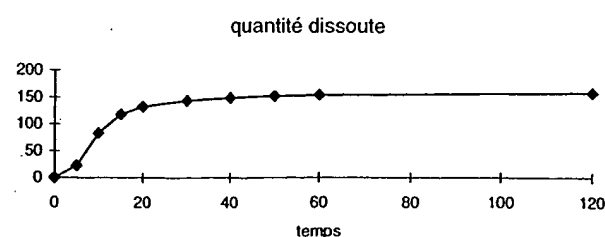
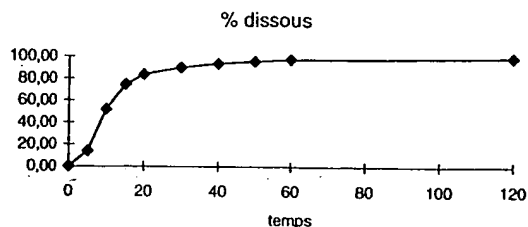
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,214 /	0,212 /	0,192 /	0,205 /	0,201 /	0,197 /
10	1000	0,775 /	0,756 /	0,761 /	0,733 /	0,724 /	0,726 /
15	1000	1,07 /	1,07 /	1,077 /	1,065 /	1,038 /	1,053 /
20	1000	1,192 /	1,173 /	1,179 /	1,198 /	1,185 /	1,198 /
30	1000	1,279 /	1,275 /	1,273 /	1,292 /	1,252 /	1,273 /
40	1000	1,316 /	1,301 /	1,3 /	1,353 /	1,329 /	1,311 /
50	1000	1,339 /	1,34 /	1,316 /	1,351 /	1,353 /	1,352 /
60	1000	1,356 /	1,357 /	1,344 /	1,384 /	1,359 /	1,369 /
120	1000	1,375 /	1,38 /	1,356 /	1,412 /	1,39 /	1,387 /

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	14,20	14,98	14,82	13,41	14,29	13,95	13,72	0,62	4,36
10,0	52,10	54,34	52,91	53,23	51,17	50,33	50,63	1,62	3,11
15,0	74,42	75,27	75,12	75,57	74,56	72,37	73,66	1,21	1,63
20,0	83,53	84,18	82,69	83,07	84,21	82,94	84,12	0,71	0,85
30,0	89,98	90,69	90,23	90,05	91,18	88,00	89,76	1,09	1,21
40,0	93,52	93,73	92,49	92,38	95,88	93,78	92,85	1,30	1,39
50,0	95,62	95,80	95,67	93,95	96,21	95,91	96,17	0,84	0,88
60,0	97,46	97,46	97,33	96,37	98,98	96,79	97,82	0,91	0,93
120,0	99,46	99,27	99,41	97,67	101,41	99,42	99,55	1,19	1,19
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	22,61	23,78	23,56	21,33	22,78	22,33	21,89	0,95	4,20
10,0	82,98	86,23	84,12	84,66	81,56	80,56	80,78	2,34	2,82
15,0	118,55	119,44	119,43	120,20	118,85	115,85	117,51	1,60	1,35
20,0	133,06	133,59	131,47	132,13	134,22	132,76	134,21	1,14	0,85
30,0	143,33	143,92	143,45	143,23	145,33	140,86	143,21	1,45	1,01
40,0	148,97	148,74	147,05	146,93	152,83	150,11	148,14	2,23	1,49
50,0	152,31	152,03	152,10	149,43	153,36	153,52	153,42	1,56	1,02
60,0	155,24	154,66	154,74	153,28	157,78	154,93	156,06	1,53	0,98
120,0	158,42	157,52	158,05	155,36	161,66	159,13	158,82	2,07	1,31
0,0									
0,0									
0,0									



durée 15 hg.1. Préparation du milieu de dissolution

- pesée de l'eau Bal GAL 014

Tare = 2,740 hg. M 15.04.97 &amp; 15/04/97

Brut = 25,490 hg M 15.04.97 &amp; 15/04/97

Net = 25,490 - 2,740 = 22,750 hg.

- pesée du LSNa Bal GAL 014

pour un milieu à 0,025 N  $\Rightarrow$ Masse de LSNa =  $22,750 \times 0,025 \times 288,6 = 164,0$  g.

Tare = 153,3 g. plus R.A.Z M 15.04.97 &amp; 15/04/97

Net = 164,0 g M 15.04.97 CC 15/04/97

2. Remplissage des bts de dissolution Bal GAL III.1 litre LSNa 0,025 N  $\Rightarrow$  1001,0 g.

bt	remise à zero	masse LSNa 0,025 N	Vérification
1	oui	1001,1 g	CC 15/04/97 M 15.04.97
2	oui	1001,1 g	CC 15/04/97 M 15.04.97
3	oui	1001,0 g	CC 15/04/97 M 15.04.97
4	oui	1001,0 g	CC 15/04/97 M 15.04.97
5	oui	1001,0 g	CC 15/04/97 M 15.04.97
6	oui	1001,0 g	CC 15/04/97 M 15.04.97

ConditionsT =  $37^{\circ}\text{C} \pm 0,5$  & 15/04/97 M 15.04.97

O 75 TPN &amp; 15/04/97 M 15.04.97

3. Revue des comprimés Boal GAL 205

*M*

15.04.97	08:26:00	1	0.0 mg
Code	2401.01-15	2	697.7 mg
ID		3	0.0 mg
		4	694.4 mg
		5	0.0 mg
		6	695.3 mg
			0.0 mg
			694.7 mg
			0.0 mg
			692.4 mg
			0.0 mg
			699.1 mg

4. lecture

chronométré GAL 124

LF 178 TER LOT RG 2401/01 15 KG

04-15-1997 12:27

*M* Lambda No. Valeur\_E

1 290.0 1 -0.0000\_1  
 2 290.0 2 0.0025\_1  
 3 290.0 *M* 3 -0.0000\_1

1 AZ Air/Air  
 2 LSNr / LSNr  
 3 AZ Air/Air

290.0	$T_0$	1	0.0004_1	290.0	$T_{30'}$	31	1.2868_1
290.0		2	0.0003_1	290.0		32	1.2690_1
290.0		3	0.0006_1	290.0		33	1.2777_1
290.0		4	-0.0004_1	290.0		34	1.2792_1
290.0		5	0.0004_1	290.0		35	1.2598_1
290.0		6	0.0001_1	290.0		36	1.2810_1
290.0	$T_{5'}$	7	0.2838_1	290.0	$T_{40'}$	37	1.3215_1
290.0		8	0.3080_1	290.0		38	1.3011_1
290.0		9	0.2877_1	290.0		39	1.2980_1
290.0		10	0.3088_1	290.0		40	1.3240_1
290.0		11	0.3204_1	290.0		41	1.2958_1
290.0		12	0.2766_1	290.0		42	1.3176_1
290.0	$T_{10'}$	13	0.8679_1	290.0	$T_{50'}$	43	1.3487_1
290.0		14	0.9082_1	290.0		44	1.3334_1
290.0		15	0.8899_1	290.0		45	1.3217_1
290.0		16	0.9083_1	290.0		46	1.3394_1
290.0		17	0.9283_1	290.0		47	1.3234_1
290.0		18	0.8616_1	290.0		48	1.3397_1
290.0	$T_{15'}$	19	1.1072_1	290.0	$T_{60'}$	49	1.3646_1
290.0		20	1.0963_1	290.0		50	1.3422_1
290.0		21	1.1043_1	290.0		51	1.3492_1
290.0		22	1.0946_1	290.0		52	1.3536_1
290.0		23	1.1126_1	290.0		53	1.3414_1
290.0		24	1.1039_1	290.0		54	1.3716_1
290.0	$T_{20'}$	25	1.2099_1	290.0	$T_{120'}$	55	1.3978_1
290.0		26	1.1839_1	290.0		56	1.3690_1
290.0		27	1.1886_1	290.0		57	1.4059_1
290.0		28	1.1801_1	290.0		58	1.3938_1
290.0		29	1.1997_1	290.0		59	1.3874_1
290.0		30	1.1975_1	290.0		60	1.4094_1

FOURNIER 1001648

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RCOS.07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 15/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2401/01 à 15 KG  
N° CAHIER : LF 178 TER n°1 p 145  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2401RG01 15 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	697,70	694,40	695,30	694,70	692,40	699,10
quantité de principe actif	160,76	160,00	160,21	160,07	159,54	161,08

Témoin 100mg/l 0,900

FOURNIER 1001649

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## SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,284	0,308	0,288	0,309	0,32	0,277
10	1000	0,868	0,908	0,89	0,908	0,928	0,862
15	1000	1,107	1,096	1,104	1,095	1,113	1,104
20	1000	1,21	1,184	1,189	1,18	1,2	1,198
30	1000	1,287	1,269	1,278	1,279	1,26	1,281
40	1000	1,322	1,301	1,298	1,324	1,296	1,318
50	1000	1,349	1,333	1,322	1,335	1,323	1,34
60	1000	1,365	1,342	1,349	1,354	1,341	1,372
120	1000	1,398	1,369	1,406	1,394	1,387	1,409

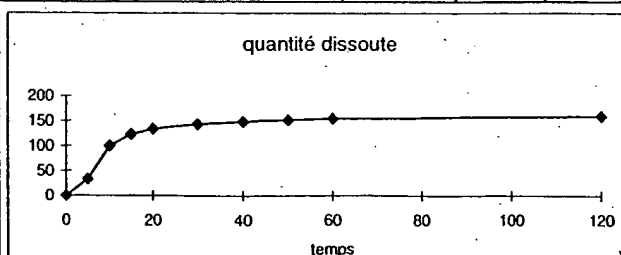
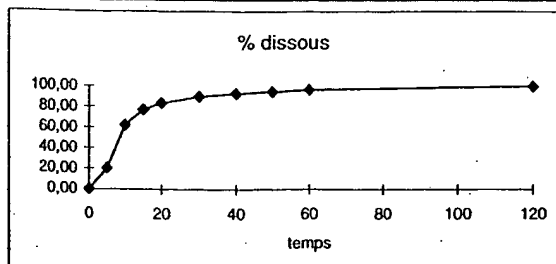
## RESULTATS EN % DISSOUS

à erreur de transmission dans une cellule significative

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	20,64	19,63	21,39	19,97	21,45	22,29	19,11	1,24	6,03
10,0	62,09	60,09	63,16	61,83	63,14	64,74	59,55	1,99	3,20
15,0	76,89	76,91	76,53	76,98	76,43	77,95	76,54	0,56	0,73
20,0	83,53	84,41	83,03	83,25	82,71	84,40	83,41	0,71	0,85
30,0	89,64	90,15	89,34	89,84	89,99	88,99	89,55	0,43	0,48
40,0	92,45	93,01	92,00	91,67	93,56	91,94	92,54	0,73	0,78
50,0	94,62	95,34	94,68	93,78	95,13	94,27	94,51	0,57	0,60
60,0	96,42	96,91	95,76	96,11	96,57	95,98	97,18	0,56	0,58
120,0	99,66	99,66	98,10	100,54	99,81	99,66	100,21	0,84	0,84
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	33,07	31,56	34,22	32,00	34,33	35,56	30,78	1,89	5,70
10,0	99,50	96,60	101,06	99,05	101,06	103,29	95,93	2,85	2,86
15,0	123,24	123,64	122,45	123,32	122,34	124,36	123,30	0,75	0,61
20,0	133,89	135,70	132,84	133,38	132,40	134,65	134,36	1,24	0,92
30,0	143,68	144,93	142,94	143,93	144,05	141,98	144,25	1,05	0,73
40,0	148,18	149,53	147,20	146,86	149,76	146,68	149,07	1,42	0,96
50,0	151,65	153,27	151,48	150,25	152,28	150,40	152,24	1,18	0,78
60,0	154,54	155,79	153,22	153,98	154,58	153,13	156,54	1,39	0,90
120,0	159,74	160,22	156,97	161,07	159,77	158,99	161,42	1,62	1,01
0,0									
0,0									
0,0									



BL09 07.97

146  
15/04/97

Gelules lipidil Micro  
lot 51

1. Préparation du milieu de dissolution

voir préparation page 143

2. Remplissage des bols de dissolution

balance BAL III. 1 litre de LNa 0,9257  $\Rightarrow$  1001,0 g.

bol	remise à zéro	pesée	vérificateur
1	oui	1001,0 g	sz 15/04/97 CC 15/04/97
2	oui	1001,0 g	sz 15/04/97 CC 15/04/97
3	oui	1001,0 g	sz 15/04/97 CC 15/04/97
4	oui	1001,0 g	sz 15/04/97 CC 15/04/97
5	oui	1001,0 g	sz 15/04/97 CC 15/04/97
6	oui	1001,0 g	sz 15/04/97 CC 15/04/97

Conditions

CC 15/04/97  
M B. Ch. 47

Température :  $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$ .  
Vitesse : 75 TPN.

Dissolvant Prolabo BAL 103  $\lambda = 290$

UV 930 BAL 108 cuve 2 mm

prélèvement de 5 ml de milieu et  
remplacement par 5 ml de milieu neuf.

1. pesée des gélules

examine 10 gélules

15.04.97 10:28:55  
Code 51

1	0.0 mg	426.3 mg
2	0.0 mg	427.2 mg
3	0.0 mg	421.8 mg
4	0.0 mg	422.3 mg
5	0.0 mg	427.9 mg
6	0.0 mg	420.4 mg

4. lectures

GELULES LIPIDIL MICRO CANADA LOT 51 LSNA 0,025M 75 TPM

15-04-1996 13:10

Lambda	No.	Valeur_E
290.0	1	-0.0018_1
290.0	2	-0.0023_1
290.0	3	-0.0027_1
290.0	4	-0.0028_1
290.0	5	-0.0028_1
290.0	6	-0.0026_1
290.0	7	0.1087_1
290.0	8	0.0785_1
290.0	9	0.0430_1
290.0	10	0.0681_1
290.0	11	0.1005_1
290.0	12	0.1173_1
290.0	13	0.4292_1
290.0	14	0.5910_1
290.0	15	0.2679_1
290.0	16	0.3941_1
290.0	17	0.3567_1
290.0	18	0.4525_1
290.0	19	0.8085_1
290.0	20	0.9199_1
290.0	21	0.6734_1
290.0	22	0.7821_1
290.0	23	0.7499_1
290.0	24	0.8261_1
290.0	25	0.9980_1
290.0	26	1.0460_1
290.0	27	0.9258_1
290.0	28	0.9971_1
290.0	29	0.9711_1

Rq : prélèvements  
au temps 60 min  
oubliés

FOURNIER 1001651

Highly Confidential  
Subject to  
Protective Order



15.04  
Code

ID

ID

ID

ID

ID

ID

4. lectures

GELULES LIPIDIL MICRO CANADA LOT 51 LSNA 0,025M 75 TPM

15-04-1996 13:10

Lambda	No.	Valeur_E
290.0	1	-0.0018_1
290.0	2	-0.0023_1
290.0	3	-0.0027_1
290.0	4	-0.0028_1
290.0	5	-0.0028_1
290.0	6	-0.0026_1
290.0	7	0.1087_1
290.0	8	0.0785_1
290.0	9	0.0430_1
290.0	10	0.0681_1
290.0	11	0.1005_1
290.0	12	0.1173_1
290.0	13	0.4292_1
290.0	14	0.5910_1
290.0	15	0.2679_1
290.0	16	0.3941_1
290.0	17	0.3567_1
290.0	18	0.4525_1
290.0	19	0.8085_1
290.0	20	0.9199_1
290.0	21	0.6734_1
290.0	22	0.7821_1
290.0	23	0.7499_1
290.0	24	0.8261_1
290.0	25	0.9980_1
290.0	26	1.0460_1
290.0	27	0.9258_1
290.0	28	0.9971_1
290.0	29	0.9711_1
290.0	30	1.0076_1
290.0	31	1.1638_1
290.0	32	1.2279_1
290.0	33	1.1551_1
290.0	34	1.2115_1
290.0	35	1.1863_1
290.0	36	1.2014_1
290.0	37	1.2791_1
290.0	38	1.2944_1
290.0	39	1.2705_1
290.0	40	1.3038_1
290.0	41	1.3165_1
290.0	42	1.2940_1
290.0	43	1.3477_1
290.0	44	1.3730_1
290.0	45	1.3262_1
290.0	46	1.3563_1
290.0	47	1.3664_1
290.0	48	1.3620_1
290.0	49	1.5623_1
290.0	50	1.5643_1
290.0	51	1.5206_1
290.0	52	1.5505_1
290.0	53	1.5851_1
290.0	54	1.5317_1

Rq : prélevements  
au temps 60 min  
oubliés

AC 09.07.97

FOURNIER 1001652

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Subject to  
Protective Order

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : C.COGET  
DATE : 15/04/97  
APPAREIL : gal 103 108 /  
LONGUEUR D'ONDE : 290 nm /  
CUVE en mm : 2mm /

TITRE : gélules lip200 M Canada lot 51  
N° CAHIER : LJ 178ter dissolution n°1 p146 /  
FICHIER : m:\commun\glnq\donnbase\l178ter\dissolution\lot 51 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM /

FOURNIER 1001653

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Protective Order

## PREPARATION DES ECHANTILLONS

masse théorique 200  
dosage théorique 200 en mg /

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

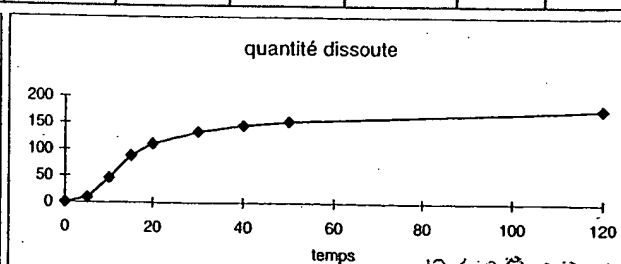
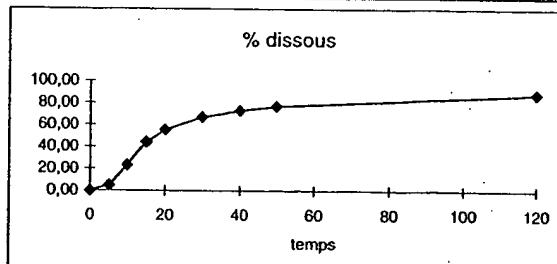
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,109 /	0,079 /	0,043 /	0,068 /	0,101 /	0,117 /
10	1000	0,429 /	0,591 /	0,268 /	0,394 /	0,357 /	0,453 /
15	1000	0,809 /	0,92 /	0,673 /	0,782 /	0,75 /	0,826 /
20	1000	0,998 /	1,046 /	0,926 /	0,997 /	0,971 /	1,008 /
30	1000	1,164 /	1,228 /	1,155 /	1,212 /	1,186 /	1,201 /
40	1000	1,279 /	1,294 /	1,271 /	1,304 /	1,317 /	1,294 /
50	1000	1,348 /	1,373 /	1,326 /	1,356 /	1,366 /	1,362 /
120	1000	1,562 /	1,564 /	1,521 /	1,551 /	1,585 /	1,532 /

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	4,79	6,06	4,39	2,39	3,78	5,61	6,50	1,56	32,56
10,0	23,10	23,86	32,86	14,90	21,91	19,86	25,20	5,99	25,93
15,0	44,21	45,09	51,30	37,48	43,57	41,79	46,05	4,60	10,41
20,0	55,42	55,82	58,55	51,72	55,73	54,28	56,39	2,28	4,12
30,0	66,80	65,32	68,95	64,70	67,96	66,49	67,39	1,61	2,42
40,0	72,81	72,03	72,96	71,46	73,40	74,10	72,89	0,95	1,30
50,0	76,61	76,22	77,71	74,87	76,65	77,19	77,03	0,99	1,29
120,0	87,95	88,48	88,70	86,07	87,86	89,74	86,85	1,33	1,51
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	9,57	12,11	8,78	4,78	7,56	11,22	13,00	3,12	32,56
10,0	46,20	47,73	65,71	29,80	43,82	39,72	50,40	11,98	25,93
15,0	88,43	90,19	102,59	74,95	87,15	83,59	92,09	9,21	10,41
20,0	110,83	111,64	117,11	103,44	111,47	108,56	112,78	4,56	4,12
30,0	133,60	130,64	137,91	129,39	135,91	132,99	134,78	3,23	2,42
40,0	145,62	144,06	145,92	142,93	146,81	148,20	145,78	1,89	1,30
50,0	153,22	152,44	155,42	149,74	153,31	154,38	154,06	1,98	1,29
120,0	175,90	176,96	177,41	172,15	175,73	179,47	173,70	2,65	1,51
0,0									
0,0									
0,0									
0,0									



RC09.07.97

Lot RG 2401/01

durée 20 h

1. Préparation du milieu de dissolution

Voir page 143

2. Remplissage des bts de dissolution Bal GAL 111

1 Ltr LNa 0,025 N  $\rightarrow$  1001,0 g.

bt	remise à zero	masse LNa 0,025 N	Vérificateur
1	oui	1001,1 g	se 15/04/97 M. S. ch. 97
2	oui	1001,0 g	se 15/04/97 M. S. ch. 97
3	oui	1001,1 g	se 15/04/97 M. S. ch. 97
4	oui	1001,0 g	se 15/04/97 M. S. ch. 97
5	oui	1001,0 g	se 15/04/97 M. S. ch. 97
6	oui	1001,0 g	se 15/04/97 M. S. ch. 97

Conditions

7 $^{\circ}$  37 $^{\circ}$ C  $\pm$  0,5

75 RPM

cc 15/04/97 M. S. ch. 97

cc 15/04/97 M. S. ch. 97

3. Pesée des comprimés Bal GAL 205

15.04.97	13:29:01					
Code	2401.01-20					
ID	1	0.0 mg	2	0.0 mg	3	0.0 mg
		695.0 mg		694.5 mg		694.0 mg
ID				0.0 mg	4	0.0 mg
				693.5 mg		695.0 mg
ID					5	0.0 mg
						695.0 mg
ID						6
						0.0 mg
						694.7 mg

FOURNIER 1001654  
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RC 09.07.97

h. Lecture

chronometre GAL AL

LF 178 TER LOT RG 2401/01 20 KG

04-15-1997 16:07

M

Lambda No. Valeur\_E

1 290.0 1 -0.0000\_1  
2 290.0 2 0.0032\_1  
3 290.0 M 3 0.0001\_1

1 AZ ALH / ALH

2 LGNa / H-SNa

290.0	T <sub>B</sub>	1	0.0003_1	290.0	T <sub>B</sub>	31	1.2522_1
290.0		2	-0.0000_1	290.0		32	1.2650_1
290.0		3	-0.0003_1	290.0		33	1.2379_1
290.0		4	-0.0003_1	290.0		34	1.2369_1
290.0		5	-0.0006_1	290.0		35	1.2660_1
290.0		6	-0.0008_1	290.0		36	1.2305_1
290.0		7	0.1860_1	290.0	T <sub>B</sub>	37	1.3017_1
290.0	T <sub>B</sub>	8	0.1807_1	290.0		38	1.2970_1
290.0		9	0.1780_1	290.0		39	1.3000_1
290.0		10	0.2085_1	290.0		40	1.3062_1
290.0		11	0.1702_1	290.0		41	1.3191_1

FOURNIER 1001655

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RC 09.07.97

N. Lechère

chronomètre GAL 124

LF 178 TER LOT RG 2401/01 20 KG

04-15-1997 16:07

M

Lambda No. Valeur\_E

1 290.0 1 -0.0000\_1  
2 290.0 2 0.0032\_1  
3 290.0 M 3 0.0001\_1

1 AZ ALH / ALH

2 L<sub>Na</sub> / L<sub>Na</sub>

3 AZ L<sub>Na</sub> / L<sub>Na</sub>

290.0	T <sub>0</sub>	1	0.0003_1	290.0	T <sub>30</sub>	31	1.2522_1
290.0		2	-0.0000_1	290.0	T <sub>30</sub>	32	1.2650_1
290.0		3	-0.0003_1	290.0		33	1.2379_1
290.0		4	-0.0003_1	290.0		34	1.2369_1
290.0		5	-0.0006_1	290.0		35	1.2660_1
290.0		6	-0.0008_1	290.0		36	1.2305_1
290.0		7	0.1860_1	290.0	T <sub>40</sub>	37	1.3017_1
290.0	T <sub>5</sub>	8	0.1807_1	290.0		38	1.2970_1
290.0		9	0.1780_1	290.0		39	1.3000_1
290.0		10	0.2085_1	290.0		40	1.3062_1
290.0		11	0.1792_1	290.0		41	1.3191_1
290.0		12	0.1966_1	290.0		42	1.3013_1
290.0	T <sub>10</sub>	13	0.6626_1	290.0	T <sub>50</sub>	43	1.3353_1
290.0		14	0.6468_1	290.0		44	1.3226_1
290.0		15	0.6328_1	290.0		45	1.3262_1
290.0		16	0.7054_1	290.0		46	1.3304_1
290.0		17	0.6616_1	290.0		47	1.3412_1
290.0		18	0.6681_1	290.0		48	1.3355_1
290.0		19	1.0178_1	290.0	T <sub>60</sub>	49	1.3548_1
290.0	T <sub>15</sub>	20	1.0080_1	290.0		50	1.3577_1
290.0		21	0.9996_1	290.0		51	1.3331_1
290.0		22	1.0317_1	290.0		52	1.3467_1
290.0		23	1.0085_1	290.0		53	1.3679_1
290.0		24	1.0033_1	290.0		54	1.3472_1
290.0	T <sub>20</sub>	25	1.1650_1	290.0	T <sub>70</sub>	55	1.3999_1
290.0		26	1.1555_1	290.0		56	1.3975_1
290.0		27	1.1507_1	290.0		57	1.3859_1
290.0		28	1.1628_1	290.0	M	58	1.3825_1
290.0		29	1.1448_1	290.0		59	1.4069_1
290.0		30	1.1368_1	290.0		60	1.3907_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
DATE : 15/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2401/01 à 20 KG  
N° CAHIER : LF 178 TER n°1 p 150  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2401RG01 20 kg  
ELUANT : L<sub>Na</sub> 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	695,00	694,50	694,00	693,50	695,80	694,70
quantité de principe actif	160,14	160,02	159,91	159,79	160,32	160,07

FOURNIER 1001656

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Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,186	0,181	0,178	0,209	0,179	0,197
10	1000	0,663	0,647	0,633	0,705	0,662	0,668
15	1000	1,018	1,008	1	1,032	1,009	1,003
20	1000	1,165	1,156	1,151	1,163	1,145	1,137
30	1000	1,252	1,265	1,238	1,237	1,266	1,231
40	1000	1,302	1,297	1,3	1,306	1,319	1,301
50	1000	1,335	1,323	1,326	1,33	1,341	1,336
60	1000	1,355	1,358	1,333	1,347	1,368	1,347

290.0 29 1.1448\_1 290.0 59 1.4069\_1  
290.0 30 1.1368\_1 290.0 60 1.3907\_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 15/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2401/01 à 20 KG  
N° CAHIER : LF 178 TER n°1 p 150  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2401RG01 20 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	695,00	694,50	694,00	693,50	695,80	694,70
quantité de principe actif	160,14	160,02	159,91	159,79	160,32	160,07

Témoin 100mg/l 0,900

Fournier 1001657

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### SUIVI DE LA DISSOLUTION

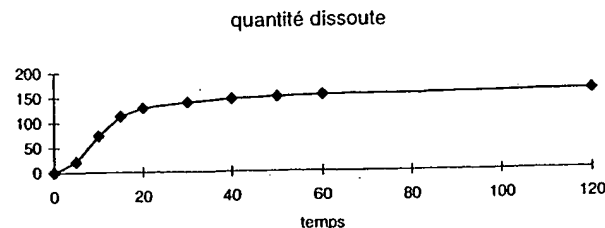
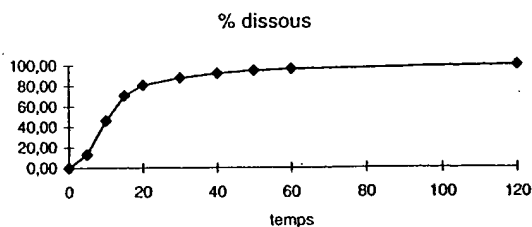
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,186	0,181	0,178	0,209	0,179	0,197
10	1000	0,663	0,647	0,633	0,705	0,662	0,668
15	1000	1,018	1,008	1	1,032	1,009	1,003
20	1000	1,165	1,156	1,151	1,163	1,145	1,137
30	1000	1,252	1,265	1,238	1,237	1,266	1,231
40	1000	1,302	1,297	1,3	1,306	1,319	1,301
50	1000	1,335	1,323	1,326	1,33	1,341	1,336
60	1000	1,355	1,358	1,333	1,347	1,368	1,347
120	1000	1,4	1,398	1,386	1,383	1,407	1,391

### RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	13,08	12,91	12,57	12,37	14,53	12,41	13,67	0,86	6,60
10,0	46,10	46,07	44,99	44,05	49,09	45,94	46,44	1,71	3,70
15,0	70,53	70,93	70,28	69,77	72,08	70,22	69,92	0,86	1,21
20,0	80,68	81,48	80,90	80,61	81,55	79,99	79,57	0,79	0,98
30,0	87,70	87,92	88,87	87,05	87,10	88,78	86,49	0,98	1,12
40,0	92,02	91,82	91,53	91,79	92,32	92,89	91,78	0,50	0,54
50,0	94,40	94,57	93,79	94,05	94,45	94,87	94,66	0,40	0,43
60,0	96,21	96,42	96,68	94,99	96,09	97,21	95,89	0,76	0,79
120,0	99,66	100,01	99,93	99,14	99,06	100,38	99,41	0,53	0,53
0,0									
0,0									
0,0									

### RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	20,93	20,67	20,11	19,78	23,22	19,89	21,89	1,37	6,53
10,0	73,77	73,77	71,99	70,43	78,45	73,66	74,33	2,70	3,66
15,0	112,88	113,58	112,46	111,56	115,17	112,58	111,93	1,32	1,17
20,0	129,13	130,48	129,46	128,90	130,30	128,25	127,37	1,20	0,93
30,0	140,36	140,80	142,22	139,20	139,17	142,33	138,45	1,67	1,19
40,0	147,28	147,05	146,48	146,78	147,53	148,92	146,91	0,88	0,60
50,0	151,07	151,44	150,09	150,39	150,92	152,10	151,52	0,76	0,50
60,0	153,98	154,40	154,71	151,90	153,55	155,85	153,49	1,34	0,87
120,0	159,49	160,15	159,91	158,53	158,29	160,94	159,12	1,02	0,64
0,0									
0,0									
0,0									



durée 15 hg.1. Préparation du milieu de dissolution

• pesée de l'eau Bal GAL 014

Tare = 2,730 hg M 16.ch.97 CC 16/04/97

Brut = 30,360 hg M 16.ch.97 CC 16/04/97

Net = 30,360 - 2,730 = 27,630 Kg

• pesée du L3Na Bal GAL 011

Pour un milieu à 0,025N → Masse L3Na à peser =

 $27,630 \times 0,025 \times 288,4 = 199,2 \text{ g.}$ Ln° PESEE SIMPLE

Date de la pesée : 1997/04/16 08:13:01



\*ARR1768 \*

PRODUIT => NALAUSF  
 POIDS NET => 0.199 KG  
 TARE => 0.153 KG  
 POIDS BRUT => 0.353 KG

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2. Remplissage des bds de dissolution bal GAL 111

Masse L3Na 0,025N → 1001,0g.

bdl	remise à zéro	masse L3Na 0,025N	Vérification
1	oui	1001,0g	CC 16/04/97 M 16.ch.97
2	oui	1001,0g	CC 16/04/97 M 16.ch.97
3	oui	1001,0g	CC 16/04/97 M 16.ch.97
4	oui	1001,1g	CC 16/04/97 M 16.ch.97
5	oui	1001,0g	CC 16/04/97 M 16.ch.97
6	oui	1001,0g	CC 16/04/97 M 16.ch.97

Conclusion:

T = 37°C ± 0,5 M 16.ch.97 CC 16.04.97

RC 09 07 97  
 M 16.ch.97  
 CC 16.04.97  
 0: 75TPN

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3. Pesée des comprimés Bal GAL 205

16,04,97	09:50:32	1		2		3		4		5		6
Code	2402.01-15											
ID		0.0 mg		0.0 mg		0.0 mg		0.0 mg		0.0 mg		0.0 mg
		696.5 mg		690.1 mg		694.3 mg		690.1 mg		693.1 mg		684.4 mg

h. Lecture

chromomètre GAL 124

LF 178 TER LOT RG 2402/01 15 KG

04-16-1997 12:28

			Lambda	No.	Valeur_E					
			290.0	T <sub>0'</sub>	1	0.0004_1	290.0	T <sub>30'</sub>	31	1.2787_1
290.0	1	0.0000_1	290.0		2	-0.0002_1	290.0		32	1.2741_1
290.0	2	0.0024_1	290.0		3	0.0003_1	290.0		33	1.2380_1
290.0	3	-0.0000_1	290.0		4	-0.0004_1	290.0		34	1.2443_1
			290.0		5	-0.0007_1	290.0		35	1.2840_1
			290.0		6	-0.0005_1	290.0		36	1.2492_1
			290.0	T <sub>5'</sub>	7	0.2931_1	290.0	T <sub>40'</sub>	37	1.3377_1
			290.0		8	0.3537_1	290.0		38	1.3029_1
			290.0		9	0.3054_1	290.0		39	1.2687_1
			290.0		10	0.3773_1	290.0		40	1.2779_1
			290.0		11	0.3351_1	290.0		41	1.3140_1
			290.0		12	0.3895_1	290.0		42	1.2874_1
			290.0	T <sub>10'</sub>	13	0.8917_1	290.0	T <sub>50'</sub>	43	1.3709_1
			290.0		14	0.9418_1	290.0		44	1.3234_1
			290.0		15	0.9193_1	290.0		45	1.3001_1
			290.0		16	0.9331_1	290.0		46	1.3164_1
			290.0		17	0.9341_1	290.0		47	1.3333_1
			290.0		18	0.9371_1	290.0		48	1.3079_1
			290.0	T <sub>15'</sub>	19	1.1103_1	290.0	T <sub>60'</sub>	49	1.3725_1
			290.0		20	1.1174_1	290.0		50	1.3368_1
			290.0		21	1.0766_1	290.0		51	1.3252_1
			290.0		22	1.1093_1	290.0		52	1.3271_1
			290.0		23	1.1190_1	290.0		53	1.3413_1
			290.0		24	1.0889_1	290.0		54	1.3102_1
			290.0	T <sub>20'</sub>	25	1.2145_1	290.0	T <sub>120'</sub>	55	1.4197_1
			290.0		26	1.1888_1	290.0		56	1.3620_1
			290.0		27	1.1684_1	290.0		57	1.3672_1
			290.0		28	1.1960_1	290.0		58	1.3710_1
			290.0		29	1.2154_1	290.0		59	1.4019_1
			290.0		30	1.1688_1	290.0		60	1.3494_1

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RC 09.07.97



# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 16/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2402/01 à 15 KG  
N° CAHIER : LF 178 TER n°1 p 153  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2402RG01 15 kg  
ELUANT : LNa 0,025 M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	696,50	690,10	694,30	690,10	693,10	684,40
quantité de principe actif	160,48	159,01	159,98	159,01	159,70	157,70

Témoin 100mg/l 0,900

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## SUIVI DE LA DISSOLUTION

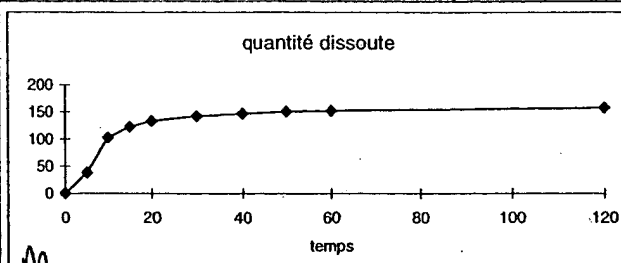
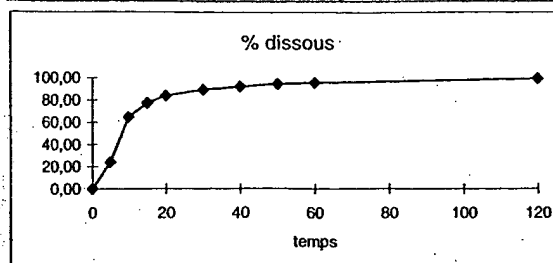
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,293	0,354	0,305	0,377	0,335	0,39
10	1000	0,892	0,942	0,919	0,933	0,934	0,937
15	1000	1,11	1,117	1,077	1,109	1,119	1,089
20	1000	1,215	1,189	1,168	1,196	1,215	1,169
30	1000	1,279	1,274	1,238	1,244	1,284	1,249
40	1000	1,338	1,303	1,269	1,278	1,314	1,287
50	1000	1,371	1,323	1,3	1,316	1,333	1,308
60	1000	1,373	1,337	1,325	1,327	1,341	1,31
120	1000	1,42	1,362	1,367	1,371	1,402	1,349

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	23,89	20,29	24,74	21,18	26,34	23,31	27,48	2,84	11,88
10,0	64,72	61,86	65,95	63,93	65,33	65,10	66,16	1,61	2,48
15,0	77,41	77,26	78,51	75,23	77,95	78,30	77,20	1,19	1,54
20,0	83,96	84,92	83,93	81,92	84,42	85,36	83,22	1,25	1,49
30,0	89,21	89,77	90,28	87,19	88,19	90,59	89,27	1,30	1,46
40,0	92,22	94,29	92,75	89,77	91,00	93,12	92,38	1,61	1,74
50,0	94,56	97,04	94,61	92,37	94,10	94,90	94,32	1,51	1,59
60,0	95,74	97,66	96,05	94,55	95,33	95,92	94,92	1,10	1,15
120,0	99,20	101,38	98,26	97,93	98,87	100,63	98,13	1,45	1,47
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	38,04	32,56	39,33	33,89	41,89	37,22	43,33	4,30	11,31
10,0	103,10	99,27	104,86	102,28	103,88	103,96	104,33	2,06	2,00
15,0	123,32	123,99	124,83	120,35	123,95	125,04	121,74	1,87	1,51
20,0	133,76	136,28	133,45	131,06	134,23	136,33	131,23	2,32	1,74
30,0	142,13	144,06	143,56	139,48	140,23	144,67	140,77	2,22	1,56
40,0	146,92	151,33	147,49	143,62	144,70	148,72	145,69	2,84	1,93
50,0	150,64	155,74	150,43	147,76	149,63	151,56	148,73	2,82	1,87
60,0	152,53	156,72	152,72	151,26	151,59	153,19	149,68	2,39	1,57
120,0	158,05	162,71	156,24	156,67	157,21	160,71	154,74	3,02	1,91
0,0									
0,0									
0,0									



Reçu 07.9.

154

16/04/97

Gelules lipidil Niro  
lot 51  
(deuxième dissolution)

1. Préparation du milieu de dissolution  
voir page 145 151 CC 16/04/97

2. Remplissage des bols de dissolution  
balance GAL III -

1 litre de LNa 0,025 N  $\Rightarrow$  1001,0 g.

bol	remise à zéro	pesée	vérificateur
1	oui	1001,0 g	M 16.04.97 CC 16/4/97
2	oui	1001,0 g	M 16.04.97 CC 16/4/97
3	oui	1001,1 g	M 16.04.97 CC 16/4/97
4	oui	1001,0 g	M 16.04.97 CC 16/4/97
5	oui	1001,1 g	M 16.04.97 CC 16/4/97
6	oui	1001,0 g	M 16.04.97 CC 16/4/97

Conditions: UV 930 GAL 108  $\lambda = 290$  nm  
cuve 2 mm.

Dissolutest Prolebo GAL 103

$t^{\circ}$   $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$

75 TPA.

CC 16/04/97 M 16.04.97

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RC09.07.97

3. Pese des aedules balance GA 205

## DISSOLUTION

m:\commun\glnq\traitdon\distern5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : C.COGET  
DATE : 16/04/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lip200 M Canada lot 51  
N° CAHIER : Lf 178ter dissolution n°1 p154  
FICHER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 51 Canada essai 2  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique 200  
dosage théorique 200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001662

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### SUIVI DE LA DISSOLUTION

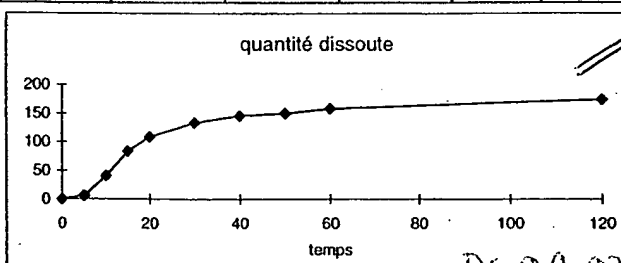
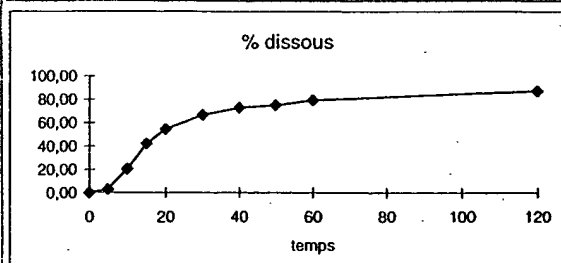
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,019	0,044	0,067	0,077	0,043	0,065
10	1000	0,252	0,304	0,43	0,397	0,355	0,439
15	1000	0,678	0,665	0,776	0,772	0,759	0,844
20	1000	0,966	0,915	0,998	0,975	0,97	1,006
30	1000	1,195	1,146	1,221	1,184	1,173	1,221
40	1000	1,305	1,25	1,318	1,297	1,287	1,322
50	1000	1,332	1,289	1,338	1,329	1,325	1,354
60	1000	1,418	1,341	1,417	1,393	1,397	1,429
120	1000	1,548	1,486	1,548	1,547	1,503	1,56

### RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	2,92	1,06	2,44	3,72	4,28	2,39	3,61	1,18	40,42
10,0	20,17	14,01	16,90	23,91	22,08	19,73	24,41	4,11	20,37
15,0	41,73	37,74	37,04	43,25	43,02	42,28	47,03	3,75	8,98
20,0	54,30	53,93	51,11	55,80	54,51	54,21	56,26	1,81	3,34
30,0	66,70	66,92	64,20	68,46	66,39	65,76	68,49	1,65	2,47
40,0	72,95	73,36	70,30	74,19	73,00	72,42	74,44	1,50	2,06
50,0	75,05	75,23	72,81	75,67	75,14	74,89	76,58	1,25	1,66
60,0	79,38	80,37	76,06	80,43	79,06	79,25	81,13	1,80	2,27
120,0	87,15	87,99	84,49	88,10	88,01	85,53	88,80	1,72	1,97
0,0									
0,0									
0,0									

### RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	5,83	2,11	4,89	7,44	8,56	4,78	7,22	2,36	40,42
10,0	40,34	28,01	33,80	47,82	44,15	39,47	48,81	8,22	20,37
15,0	83,45	75,48	74,08	86,50	86,04	84,55	94,06	7,50	8,98
20,0	108,61	107,86	102,23	111,60	109,03	108,42	112,53	3,63	3,34
30,0	133,41	133,84	128,40	136,93	132,79	131,52	136,97	3,30	2,47
40,0	145,90	146,73	140,60	148,38	146,00	144,83	148,88	3,00	2,06
50,0	150,11	150,45	145,62	151,34	150,28	149,77	153,17	2,50	1,66
60,0	158,77	160,75	152,12	160,86	158,13	158,51	162,25	3,61	2,27
120,0	174,31	175,98	168,97	176,20	176,01	171,06	177,60	3,44	1,97
0,0									
0,0									
0,0									



PL 09.07.97

3. Pesée des gélules

balance GAL 205

16.04.97	08:53:41					
Code	51					
ID	1	0.0 mg	426.6 mg	0.0 mg	429.2 mg	0.0 mg
ID	2	0.0 mg	412.7 mg	0.0 mg	418.6 mg	0.0 mg
ID	3	0.0 mg		0.0 mg		0.0 mg
ID	4	427.2 mg		429.2 mg		
ID	5					
ID	6					

4. lectures et résultats

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16-04-1996 10:28

Lambda No. Valeur\_E

290.0	1	-0.0008_1
290.0	2	-0.0030_1
290.0	3	-0.0038_1 T0
290.0	4	-0.0034_1
290.0	5	-0.0034_1
290.0	6	-0.0035_1
290.0	7	0.0194_1
290.0	8	0.0444_1
290.0	9	0.0673_1 T5
290.0	10	0.0770_1
290.0	11	0.0430_1
290.0	12	0.0651_1
290.0	13	0.2522_1
290.0	14	0.3042_1
290.0	15	0.4301_1 T10
290.0	16	0.3970_1
290.0	17	0.3548_1
290.0	18	0.4392_1
290.0	19	0.6778_1
290.0	20	0.6650_1
290.0	21	0.7762_1 T15
290.0	22	0.7722_1
290.0	23	0.7591_1
290.0	24	0.8442_1
290.0	25	0.9656_1
290.0	26	0.9149_1
290.0	27	0.9978_1
290.0	28	0.9754_1 T20
290.0	29	0.9704_1
290.0	30	1.0063_1
290.0	31	1.1953_1
290.0	32	1.1458_1
290.0	33	1.2211_1 T30
290.0	34	1.1836_1
290.0	35	1.1729_1
290.0	36	1.2210_1
290.0	37	1.3047_1
290.0	38	1.2495_1
290.0	39	1.3178_1 T40
290.0	40	1.2972_1
290.0	41	1.2869_1
290.0	42	1.3215_1
290.0	43	1.3323_1
290.0	44	1.2887_1
290.0	45	1.3380_1 T50
290.0	46	1.3291_1
290.0	47	1.3249_1
290.0	48	1.3542_1
290.0	49	1.4175_1
290.0	50	1.3407_1
290.0	51	1.4168_1 T60
290.0	52	1.3930_1
290.0	53	1.3965_1
290.0	54	1.4294_1
290.0	55	1.5479_1
290.0	56	1.4856_1
290.0	57	1.5475_1 T120
290.0	58	1.5466_1
290.0	59	1.5026_1
290.0	60	1.5599_1

FOURNIER 1001664

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16.ch.97

Lot RG 240210.1

durée 20hg.1. Préparation du milieu de dissolution

Voir page 151

2. Remplissage des bts de dissolution Bal GAZ M1 Ltr LNa 0,025N  $\rightarrow$  1001,0g.

btl	remise à zero	masse LNa 0,025N	Vérificateur
1	oui	1001,0g	CC 16/04/97 M 16.ch.97
2	oui	1001,0g	CC 16/04/97 M 16.ch.97
3	oui	1001,0g	CC 16/04/97 M 16.ch.97
4	oui	1001,0g	CC 16/04/97 M 16.ch.97
5	oui	1001,1g	CC 16/04/97 M 16.ch.97
6	oui	1001,1g	CC 16/04/97 M 16.ch.97

Conditions $T^{\circ} = 37^{\circ}\text{C} \pm 0,5$ 

CC 16/04/97 M 16.ch.97

 $\phi = 75 \text{ TPN}$ 

CC 16/04/97 M 16.ch.97

3. Pesée des comprimés Bal GAZ 205

16.04.97 Code 2402.01-20	1	2	3	4	5	6
ID	ID	ID	ID	ID	ID	ID
0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
692.3 mg	690.6 mg	693.8 mg	697.6 mg	692.6 mg	694.3 mg	

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Protective Order

RC 10.07.97

4. Lection chronometre GAL 104

LF 178 TER LOT RG 2402/01 20 KG

04-16-1997 16:05

			Lambda	No.	Valeur_E					
290.0	1	0.0000_1	290.0	1	0.0003_1	290.0	T <sub>1</sub> '	31	1.2395_1	
	2	0.0028_1		290.0	2	0.0008_1	290.0	T <sub>2</sub> '	32	1.2634_1
	3	-0.0000_1		290.0	3	0.0004_1	290.0		33	1.2678_1
290.0			290.0	4	0.0002_1	290.0		34	1.2659_1	
290.0			290.0	5	-0.0001_1	290.0		35	1.2347_1	
290.0			290.0	6	-0.0005_1	290.0		36	1.2511_1	
			290.0	7	0.2149_1	290.0	T <sub>3</sub> '	37	1.2833_1	
			290.0	8	0.2091_1	290.0	T <sub>4</sub> '	38	1.3060_1	
			290.0	9	0.2432_1	290.0		39	1.3214_1	
			290.0	10	0.2107_1	290.0		40	1.3043_1	
			290.0	11	0.2478_1	290.0		41	1.2897_1	
			290.0	12	0.2236_1	290.0		42	1.3175_1	
			290.0	13	0.7296_1	290.0	T <sub>5</sub> '	43	1.3125_1	
			290.0	14	0.7186_1	290.0		44	1.3430_1	
			290.0	15	0.7758_1	290.0		45	1.3345_1	
			290.0	16	0.7082_1	290.0		46	1.3343_1	

FOURNIER 1001666

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Protective Order

RC 10.07.97

LF 178 TER LOT RG 2402/01 20 KG

04-16-1997 16:05

M

	Lambda	No.	Valeur_E
290.0	1	0.0003_1	290.0 31 1.2395_1
290.0	2	0.0008_1	290.0 32 1.2634_1
290.0	3	0.0004_1	290.0 33 1.2678_1
290.0	4	0.0002_1	290.0 34 1.2659_1
290.0	5	-0.0001_1	290.0 35 1.2347_1
290.0	6	-0.0005_1	290.0 36 1.2511_1
290.0	7	0.2149_1	290.0 37 1.2833_1
290.0	8	0.2091_1	290.0 38 1.3060_1
290.0	9	0.2432_1	290.0 39 1.3214_1
290.0	10	0.2107_1	290.0 40 1.3043_1
290.0	11	0.2478_1	290.0 41 1.2897_1
290.0	12	0.2236_1	290.0 42 1.3175_1
290.0	13	0.7296_1	290.0 43 1.3125_1
290.0	14	0.7186_1	290.0 44 1.3430_1
290.0	15	0.7758_1	290.0 45 1.3345_1
290.0	16	0.7082_1	290.0 46 1.3343_1
290.0	17	0.7734_1	290.0 47 1.3072_1
290.0	18	0.7220_1	290.0 48 1.3227_1
290.0	19	1.0091_1	290.0 49 1.3229_1
290.0	20	1.0174_1	290.0 50 1.3584_1
290.0	21	1.0701_1	290.0 51 1.3359_1
290.0	22	1.0310_1	290.0 52 1.3382_1
290.0	23	1.0318_1	290.0 53 1.3390_1
290.0	24	1.0341_1	290.0 54 1.3702_1
290.0	25	1.1416_1	290.0 55 1.3819_1
290.0	26	1.1547_1	290.0 56 1.4108_1
290.0	27	1.1869_1	290.0 57 1.3696_1
290.0	28	1.1574_1	290.0 58 1.3925_1
290.0	29	1.1442_1	290.0 59 1.3771_1
290.0	30	1.1566_1	290.0 60 1.4079_1

M

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 16/04/97 ✓  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm ✓  
CUVE en mm : 2 ✓

TITRE : LF 178 TER RG 2402/01 à 20 KG  
N° CAHIER : LF 178 TER n°1 p 157 ✓  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2402RG01 20 kg  
ELUANT : LSNa 0,025 M ✓  
AGITATION : 75 TPM ✓

FOURNIER 1001667

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## PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg ✓

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	692,30 ✓	690,60 ✓	693,80 ✓	697,60 ✓	692,60 ✓	694,30 ✓
quantité de principe actif	159,52	159,12	159,86	160,74	159,59	159,98

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volumen en ml						
0	0	0	0	0	0	0	0
5	1000	0,215 ✓	0,209 ✓	0,243 ✓	0,211 ✓	0,248 ✓	0,224 ✓
10	1000	0,73 ✓	0,719 ✓	0,776 ✓	0,708 ✓	0,773 ✓	0,722 ✓
15	1000	1,009 ✓	1,017 ✓	1,07 ✓	1,031 ✓	1,032 ✓	1,034 ✓
20	1000	1,142 ✓	1,155 ✓	1,187 ✓	1,157 ✓	1,144 ✓	1,157 ✓
30	1000	1,24 ✓	1,263 ✓	1,268 ✓	1,266 ✓	1,235 ✓	1,251 ✓
40	1000	1,283 ✓	1,306 ✓	1,321 ✓	1,304 ✓	1,29 ✓	1,318 ✓
50	1000	1,313 ✓	1,343 ✓	1,335 ✓	1,334 ✓	1,307 ✓	1,323 ✓



# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 16/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2402/01 à 20 KG  
N° CAHIER : LF 178 TER n°1 p 157  
FICHIER : M:\commun\glnq\donnbase\Lf178ter\dissolution\lot 2402RG01 20 kg  
ELUANT : LNa 0,025 M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	692,30	690,60	693,80	697,60	692,60	694,30
quantité de principe actif	159,52	159,12	159,86	160,74	159,59	159,98

Témoin 100mg/l 0,900

FOURNIER 1001668

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Subject to  
Protective Order

## SUIVI DE LA DISSOLUTION

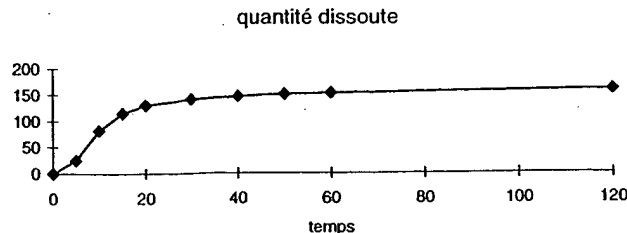
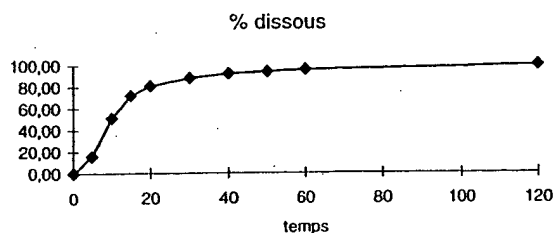
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,215	0,209	0,243	0,211	0,248	0,224
10	1000	0,73	0,719	0,776	0,708	0,773	0,722
15	1000	1,009	1,017	1,07	1,031	1,032	1,034
20	1000	1,142	1,155	1,187	1,157	1,144	1,157
30	1000	1,24	1,263	1,268	1,266	1,235	1,251
40	1000	1,283	1,306	1,321	1,304	1,29	1,318
50	1000	1,313	1,343	1,335	1,334	1,307	1,323
60	1000	1,323	1,358	1,336	1,338	1,339	1,37
120	1000	1,382	1,411	1,37	1,396	1,377	1,408

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	15,64	14,98	14,59	16,89	14,59	17,27	15,56	1,17	7,49
10,0	51,39	50,92	50,28	54,02	49,01	53,91	50,22	2,08	4,05
15,0	72,10	70,61	71,34	74,72	71,59	72,21	72,14	1,41	1,96
20,0	81,14	80,23	81,33	83,23	80,65	80,37	81,05	1,10	1,36
30,0	88,28	87,45	89,27	89,27	88,59	87,10	87,98	0,92	1,04
40,0	92,18	90,88	92,72	93,39	91,65	91,36	93,06	1,02	1,11
50,0	94,17	93,41	95,76	94,83	94,18	92,99	93,87	1,00	1,06
60,0	95,90	94,57	97,27	95,36	94,91	95,67	97,59	1,25	1,31
120,0	99,61	99,14	101,45	98,19	99,39	98,79	100,71	1,23	1,23
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	25,00	23,89	23,22	27,00	23,44	27,56	24,89	1,86	7,45
10,0	82,13	81,23	80,01	86,36	78,78	86,03	80,35	3,25	3,96
15,0	115,22	112,64	113,52	119,46	115,07	115,23	115,41	2,35	2,04
20,0	129,66	127,97	129,41	133,05	129,64	128,25	129,66	1,81	1,40
30,0	141,07	139,50	142,06	142,71	142,39	139,00	140,74	1,57	1,11
40,0	147,30	144,96	147,54	149,30	147,32	145,80	148,88	1,69	1,15
50,0	150,49	149,01	152,37	151,59	151,38	148,40	150,17	1,56	1,04
60,0	153,24	150,85	154,78	152,44	152,56	152,68	156,13	1,89	1,23
120,0	159,17	158,14	161,43	156,96	159,75	157,65	161,11	1,87	1,17
0,0									
0,0									
0,0									



6/04/97

Gelules lipidil Duno  
lot 52

# 1. Préparation du milieu de dissolution

voir page 151

# 2. Remplissage des bols de dissolution

1 litre LSNa 0,025 N  $\Rightarrow$  1001,0 g à peser

bol	remise à zéro	pesée	seri. f. cateur
1	oui	1001,0 g.	M 16.04.97 CC 16/04/97
2	oui	1001,0 g.	M 16.04.97 CC 16/04/97
3	oui	1001,0 g.	M 16.04.97 CC 16/04/97
4	oui	1001,0 g.	M 16.04.97 CC 16/04/97
5	oui	1001,0 g.	M 16.04.97 CC 16/04/97
6	oui	1001,0 g.	M 16.04.97 CC 16/04/97

Conditions : UV930 GAC 108  $\lambda = 290 \text{ nm}$

cuve 2 mm -

dissolvant Prolabo GAC 103

 $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$ 

CC 16/04/97 M 16.04.97

75 TPN

CC 16/04/97 M 16.04.97

FOURNIER 1001669

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Protective Order

RC 10.07.97

3. Pesée des gélules

balance BAL 205

16.04.97	13:56:50	52	1	0.0 mg	2	0.0 mg	3	0.0 mg	4	0.0 mg	5	0.0 mg	46	0.0 mg
Code			ID	422.3 mg	ID	424.7 mg	ID	426.2 mg	ID	424.4 mg	ID	428.1 mg	ID	424.1 mg

4. lectures

GELULES LIPIDIL MICRO CANADA LOT 52 LSNA 0,025M 75 TPM

16-04-1996 15:13

Lambda	No.	Valeur E
290.0	1	-0.0031_1
290.0	2	-0.0033_1
290.0	3	-0.0033_1 TO
290.0	4	-0.0031_1
290.0	5	-0.0029_1
290.0	6	-0.0027_1
290.0	7	0.0597_1
290.0	8	0.0373_1
290.0	9	0.0465_1
290.0	10	0.0986_1 T5
290.0	11	0.0742_1
290.0	12	0.0844_1
290.0	13	0.3238_1
290.0	14	0.3083_1
290.0	15	0.4145_1
290.0	16	0.3845_1 T10

**FOURNIER 1001671**  
**Highly Confidential**  
**Subject to**  
**Protective Order**

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : C.COGET  
 DATE : 16/04/97  
 APPAREIL : gal 103 108  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2mm

TITRE : gélules lip200 M Canada lot 52  
 N° CAHIER : Lf 178ter dissolution n°1 p158  
 FICHIER : m:\commun\glnq\donnbases\lf178ter\dissolution\lot 52 Canada  
 ELUANT : LSNa 0,025M  
 AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001672

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 Subject to  
 Protective Order

## SUIVI DE LA DISSOLUTION

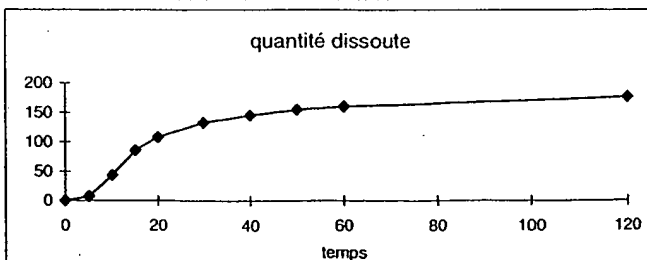
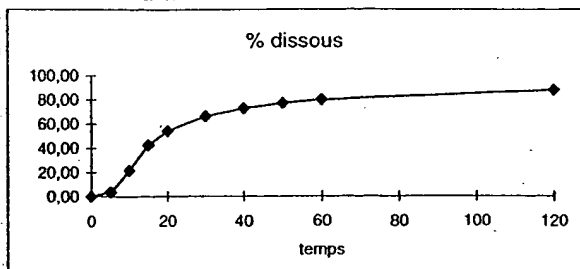
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,06	0,037	0,047	0,099	0,074	0,084
10	1000	0,324	0,308	0,415	0,385	0,439	0,465
15	1000	0,676	0,696	0,812	0,757	0,817	0,858
20	1000	0,939	0,917	0,989	0,981	0,988	1,013
30	1000	1,149	1,153	1,214	1,194	1,204	1,184
40	1000	1,231	1,28	1,311	1,303	1,323	1,311
50	1000	1,353	1,345	1,373	1,374	1,379	1,368
60	1000	1,396	1,403	1,415	1,421	1,432	1,406
120	1000	1,553	1,536	1,556	1,551	1,554	1,54

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	3,71	3,33	2,06	2,61	5,50	4,11	4,67	1,29	34,84
10,0	21,65	18,02	17,12	23,07	21,42	24,41	25,86	3,50	16,15
15,0	42,87	37,66	38,76	45,24	42,19	45,53	47,82	4,04	9,43
20,0	54,29	52,46	51,23	55,30	54,84	55,26	56,67	2,03	3,74
30,0	66,33	64,39	64,60	68,07	66,95	67,53	66,45	1,53	2,30
40,0	72,78	69,26	71,98	73,80	73,34	74,48	73,83	1,92	2,63
50,0	77,15	76,38	75,94	77,61	77,64	77,96	77,37	0,80	1,04
60,0	80,13	79,15	79,54	80,32	80,64	81,28	79,86	0,78	0,97
120,0	88,09	88,26	87,32	88,55	88,25	88,46	87,69	0,48	0,55
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	7,43	6,67	4,11	5,22	11,00	8,22	9,33	2,59	34,84
10,0	43,30	36,03	34,24	46,14	42,83	48,82	51,71	6,99	16,15
15,0	85,73	75,32	77,53	90,48	84,38	91,06	95,64	8,08	9,43
20,0	108,59	104,92	102,47	110,60	109,69	110,52	113,34	4,06	3,74
30,0	132,66	128,78	129,20	136,15	133,90	135,07	132,90	3,05	2,30
40,0	145,56	138,53	143,95	147,60	146,68	148,96	147,67	3,83	2,63
50,0	154,30	152,77	151,88	155,22	155,29	155,91	154,73	1,60	1,04
60,0	160,26	158,30	159,08	160,65	161,27	162,57	159,71	1,55	0,97
120,0	176,18	176,52	174,63	177,10	176,51	176,92	175,38	0,96	0,55
0,0									
0,0									
0,0									



RC 10.03.92

A.ch.97

Lot RG 2403101

10

date: 15 kg.1. Préparation du milieu de dissolution

- pesée de l'eau Bal GAL 01h

Tare = 2,725 kg M A.ch.97 &amp; 17/04/97

Brut = 23,830 kg M A.ch.97 &amp; 17/04/97

Net = 23,830 - 2,725 = 21,105 kg

- pesée du LNa Bal GAL 01h

Pour un milieu à 0,025N, masse de LNa à peser  
 =  $21,105 \times 0,025 \times 288,4 = 152,6 \text{ g.}$

**PESEE SIMPLE**

Date de la pesée : 1997/04/17 08:11:18



\*ARR1768\*

PRODUIT => NALAUSF  
 POIDS NET => 0.153 KG  
 TARE => 0.154 KG  
 POIDS BRUT => 0.306 KG

M

2. Remplissage des bds de dissolution Bal GAL M

Même LNa 0,025N - 1001,0g.

bdl	remise à zero	masse LNa 0,025N	Vérification
1	oui	1001,0g	CC 17/04/97 M A.ch.
2	oui	1001,0g	CC 17/04/97 M A.ch.
3	oui	1001,0g	CC 17/04/97 M A.ch.
4	oui	1001,0g	CC 17/04/97 M A.ch.
5	oui	1001,0g	CC 17/04/97 M A.ch.
6	oui	1001,0g	CC 17/04/97 M A.ch.

FOURNIER 1001673

Highly Confidential  
 Subject to  
 Protective Order

Conditions

T° 37°C ± 0,5 M A.ch.97

CC 17/04/97

② 75 TPN M A.ch.97

CC 17/04/97 M A.ch.

### 3. Pesée des comprimés Bal GAL 205

17.04.97 09:54:31  
Code 2403.01-15

1	0.0 mg
2	697.7 mg
3	0.0 mg
4	696.5 mg
5	0.0 mg
6	694.2 mg
7	0.0 mg
8	693.8 mg
9	0.0 mg
10	701.4 mg
11	0.0 mg
12	697.3 mg

### 4. Lecture Chronometre GAL 124

LF 178 TER LOT RG 2403/01 15 KG

04-17-1997 12:30

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0025_1
290.0	3	-0.0000_1
290.0	4	-0.0007_1
290.0	5	-0.0009_1
290.0	6	-0.0005_1
290.0	7	0.2685_1
290.0	8	0.2560_1
290.0	9	0.3077_1
290.0	10	0.3409_1
290.0	11	0.2577_1
290.0	12	0.2936_1
290.0	13	0.8596_1
290.0	14	0.8588_1
290.0	15	0.8988_1
290.0	16	0.9105_1
290.0	17	0.8522_1
290.0	18	0.8757_1
290.0	19	1.0998_1
290.0	20	1.1041_1
290.0	21	1.1031_1
290.0	22	1.0964_1
290.0	23	1.1116_1
290.0	24	1.0967_1
290.0	25	1.2038_1
290.0	26	1.2093_1
290.0	27	1.1869_1
290.0	28	1.1917_1
290.0	29	1.2247_1
290.0	30	1.1930_1
290.0	31	1.2947_1
290.0	32	1.2759_1
290.0	33	1.2624_1
290.0	34	1.2701_1
290.0	35	1.3034_1
290.0	36	1.3040_1
290.0	37	1.3313_1
290.0	38	1.3369_1
290.0	39	1.3168_1
290.0	40	1.3058_1
290.0	41	1.3545_1
290.0	42	1.3253_1
290.0	43	1.3604_1
290.0	44	1.3210_1
290.0	45	1.3148_1
290.0	46	1.3110_1
290.0	47	1.3879_1
290.0	48	1.3655_1
290.0	49	1.3648_1
290.0	50	1.3681_1
290.0	51	1.3311_1
290.0	52	1.3297_1
290.0	53	1.4008_1
290.0	54	1.3683_1
290.0	55	1.4110_1
290.0	56	1.4063_1
290.0	57	1.3813_1
290.0	58	1.3667_1
290.0	59	1.4250_1
290.0	60	1.4101_1

FOURNIER 1001674

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Protective Order

HC 10-07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distern5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 17/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2403/01 à 15 KG  
N° CAHIER : LF 178 TER n°1 p 163  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2403RG01 15 kg  
ELUANT : LNa 0,025 M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	697,70	696,50	694,20	693,80	701,40	697,30
quantité de principe actif	160,76	160,48	159,95	159,86	161,61	160,67

Témoin 100mg/l 0,900

FOURNIER 1001675

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## SUIVI DE LA DISSOLUTION

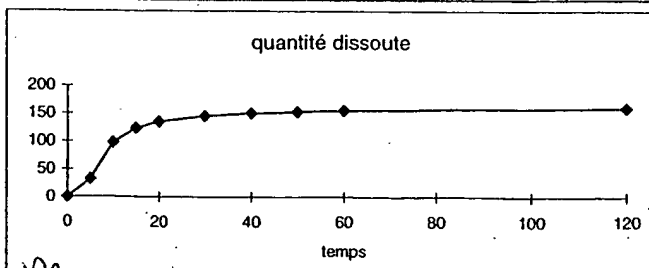
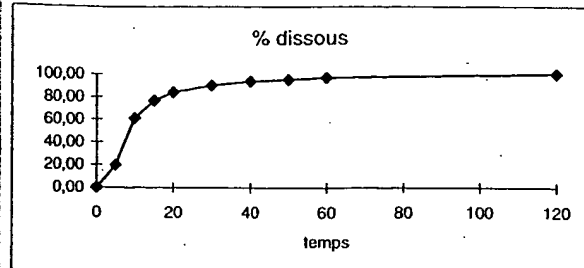
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,269	0,256	0,308	0,341	0,258	0,294
10	1000	0,86	0,859	0,899	0,911	0,852	0,876
15	1000	1,1	1,104	1,103	1,096	1,112	1,097
20	1000	1,204	1,209	1,187	1,192	1,225	1,193
30	1000	1,295	1,276	1,262	1,27	1,303	1,304
40	1000	1,331	1,337	1,317	1,306	1,355	1,325
50	1000	1,36	1,321	1,315	1,311	1,388	1,366
60	1000	1,365	1,368	1,331	1,33	1,401	1,368
120	1000	1,411	1,406	1,381	1,367	1,425	1,41

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	19,91	18,59	17,72	21,40	23,70	17,74	20,33	2,37	11,88
10,0	60,74	59,53	59,56	62,56	63,44	58,66	60,68	1,88	3,10
15,0	76,67	76,42	76,82	77,04	76,61	76,83	76,27	0,29	0,38
20,0	83,94	83,99	84,47	83,26	83,67	84,98	83,29	0,69	0,82
30,0	90,12	90,69	89,53	88,88	89,50	90,77	91,38	0,96	1,06
40,0	93,58	93,63	94,20	93,14	92,44	94,79	93,28	0,83	0,88
50,0	95,07	96,09	93,55	93,46	93,25	97,53	96,57	1,87	1,97
60,0	96,72	96,91	97,26	95,02	95,02	98,90	97,18	1,49	1,54
120,0	99,92	100,56	100,37	98,96	98,06	101,03	100,56	1,15	1,15
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	31,96	29,89	28,44	34,22	37,89	28,67	32,67	3,69	11,56
10,0	97,51	95,71	95,59	100,06	101,41	94,81	97,50	2,68	2,75
15,0	123,09	122,85	123,29	123,23	122,47	124,17	122,54	0,63	0,51
20,0	134,78	135,02	135,57	133,17	133,75	137,35	133,82	1,54	1,14
30,0	144,70	145,80	143,68	142,17	143,08	146,69	146,81	1,99	1,37
40,0	150,25	150,52	151,17	148,98	147,78	153,19	149,87	1,87	1,24
50,0	152,66	154,48	150,13	149,49	149,06	157,61	155,16	3,56	2,33
60,0	155,29	155,79	156,09	152,00	151,90	159,83	156,14	2,98	1,92
120,0	160,44	161,66	161,07	158,29	156,75	163,27	161,57	2,42	1,51
0,0									
0,0									
0,0									



27 10 1997



17/04/97

Gelules Lipidil Niro  
lot 53

1. Préparation du milieu de dissolution

voir page 161

2. Remplissage des bols de dissolution

1 litre LSNa 0,025 N  $\Rightarrow$  1001,0 g à peser

bol	remise à zéro	pesée	ser. p. cateur
1	oui	1001,0 g.	M 17.ch.97 CC 17/04/97
2	oui	1001,1 g.	M 17.ch.97 CC 17/04/97
3	oui	1001,0 g.	M 17.ch.97 CC 17/04/97
4	oui	1001,0 g.	M 17.ch.97 CC 17/04/97
5	oui	1001,0 g.	M 17.ch.97 CC 17/04/97
6	oui	1001,0 g.	M 17.ch.97 CC 17/04/97

Conditions: UV930 GAC 108  $\lambda = 290 \text{ nm}$   
cuve 2 mm.

dissolvant Proclabo GAC 103

37°C  $\pm 0,5^\circ\text{C}$  M 17.ch.97 CC 17/04/97

75 TPN M 17.ch.97 CC 17/04/97

FOURNIER 1001676

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Re 10.07.97

### 3. Pesée des gelules

Balance AB-204 GALL 205

17.04.97 09:38:35  
Code 53

1

12

3

4

5

6

ID 0.0 mg

425.6 mg

ID 0.0 mg

430.1 mg

ID 0.0 mg

417.4 mg

ID 0.0 mg

427.1 mg

ID 0.0 mg

432.1 mg

ID 0.0 mg

426.9 mg

### 4. Lecture

GELULES LIPIDIL MICRO CANADA LOT 53 LSNA 0,025M 75 TPM

17-04-1996 11:0

Lambda	No.	Valeur_E
290.0	1	-0.0026_1
290.0	2	-0.0026_1
290.0	3	-0.0027_1
290.0	4	-0.0026_1
290.0	5	-0.0028_1
290.0	6	-0.0027_1
290.0	7	0.0758_1
290.0	8	0.0804_1
290.0	9	0.0684_1
290.0	10	0.0686_1
290.0	11	0.0884_1
290.0	12	0.1666_1
290.0	13	0.3755_1
290.0	14	0.3650_1

FOURNIER 1001677  
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BC 10.07.97

FOURNIER 1001678

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Lambda	No.	Value
290.0	1	-0.0026
290.0	2	-0.0026
290.0	3	-0.0027
290.0	4	-0.0026
290.0	5	-0.0028
290.0	6	-0.0027
290.0	7	0.0758
290.0	8	0.0804
290.0	9	0.0684
290.0	10	0.0686
290.0	11	0.0884
290.0	12	0.1666
290.0	13	0.3755
290.0	14	0.3650
290.0	15	0.4813
290.0	16	0.4885
290.0	17	0.6140
290.0	18	0.7554
290.0	19	0.7536
290.0	20	0.8054
290.0	21	0.8275
290.0	22	0.7783
290.0	23	0.9043
290.0	24	0.9802
290.0	25	0.9748
290.0	26	0.9833
290.0	27	1.0107
290.0	28	1.0021
290.0	29	1.0739
290.0	30	1.0714
290.0	31	1.2002
290.0	32	1.1863
290.0	33	1.1903
290.0	34	1.2294
290.0	35	1.2256
290.0	36	1.2604
290.0	37	1.3089
290.0	38	1.3002
290.0	39	1.2827
290.0	40	1.3255
290.0	41	1.3419
290.0	42	1.3412
290.0	43	1.3869
290.0	44	1.3613
290.0	45	1.3554
290.0	46	1.3889
290.0	47	1.4014
290.0	48	1.4049
290.0	49	1.4137
290.0	50	1.4235
290.0	51	1.3808
290.0	52	1.4212
290.0	53	1.4554
290.0	54	1.4426
290.0	55	1.5502
290.0	56	1.5543
290.0	57	1.5072
290.0	58	1.5433
290.0	59	1.5836
290.0	60	1.5545

1120

1160

1150

1140

1130

1120

1115

1110

1105

1100

# DISSOLUTION

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : C.COGET  
DATE : 17/04/97  
APPAREIL : gal 103 108  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lip200 M Canada lot 53  
N° CAHIER : LI 178ter dissolution n°1 p164  
FICHIER : m:\commun\glnq\donnbase\LI178ter\dissolution\lot 53 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## PREPARATION DES ECHANTILLONS

masse théorique 200  
dosage théorique 200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001679

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## SUIVI DE LA DISSOLUTION

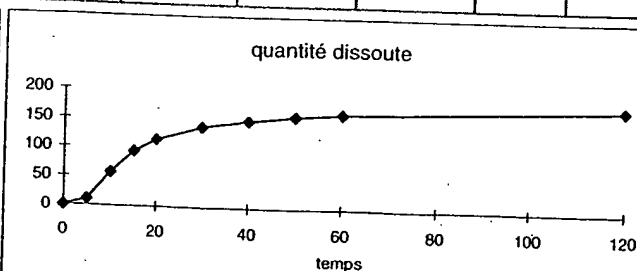
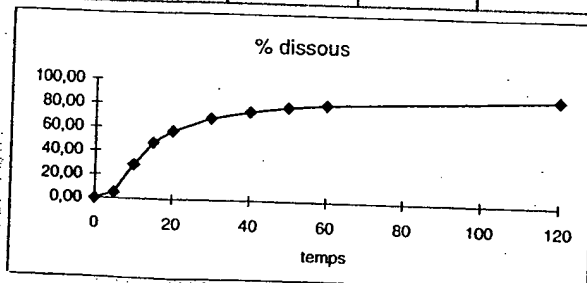
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,076	0,08	0,068	0,069	0,088	0,167
10	1000	0,376	0,365	0,481	0,489	0,614	0,755
15	1000	0,754	0,805	0,828	0,778	0,904	0,98
20	1000	0,975	0,983	1,011	1,002	1,074	1,071
30	1000	1,2	1,186	1,19	1,229	1,226	1,26
40	1000	1,309	1,3	1,283	1,326	1,342	1,341
50	1000	1,387	1,361	1,355	1,389	1,401	1,405
60	1000	1,414	1,424	1,381	1,421	1,455	1,443
120	1000	1,55	1,554	1,507	1,543	1,584	1,555

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	5,07	4,22	4,44	3,78	3,83	4,89	9,28	2,10	41,38
10,0	28,54	20,91	20,30	26,74	27,19	34,14	41,99	8,29	29,03
15,0	46,92	42,01	44,85	46,15	43,38	50,42	54,70	4,78	10,19
20,0	57,03	54,50	54,96	56,55	56,04	60,11	60,03	2,47	4,32
30,0	68,19	67,27	66,51	66,77	68,93	68,86	70,83	1,65	2,42
40,0	74,18	73,66	73,17	72,27	74,66	75,64	75,68	1,38	1,86
50,0	78,22	78,36	76,92	76,63	78,53	79,29	79,60	1,22	1,55
60,0	80,83	80,24	80,80	78,45	80,69	82,68	82,11	1,49	1,84
120,0	88,21	88,19	88,42	85,83	87,86	90,25	88,73	1,43	1,62
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	10,15	8,44	8,89	7,56	7,67	9,78	18,56	4,20	41,38
10,0	57,09	41,82	40,60	53,48	54,37	68,27	83,98	16,58	29,03
15,0	93,84	84,03	89,69	92,31	86,75	100,83	109,40	9,56	10,19
20,0	114,06	109,00	109,92	113,10	112,08	120,23	120,06	4,93	4,32
30,0	136,39	134,55	133,02	133,55	137,85	137,71	141,65	3,30	2,42
40,0	148,36	147,32	146,34	144,54	149,32	151,28	151,35	2,76	1,86
50,0	156,44	156,72	153,84	153,26	157,05	158,58	159,21	2,43	1,55
60,0	161,66	160,49	161,60	156,90	161,38	165,36	164,21	2,98	1,84
120,0	176,43	176,38	176,84	171,67	175,72	180,50	177,46	2,86	1,62
0,0									
0,0									
0,0									



RC 10.07.97

duveté 18 kg.

1. Préparation du milieu de dissolution  
Voir page 161

2. Remplissage des bûts de dissolution Bal GAL III  
1 Litré LSK 0.025N  $\rightarrow$  1001.0 g.

bûl	remise à zero	masse LSK 0.025N	Vérificateur
1	oui	1001.0g	CC 17/04/97 M. Mc
2	oui	1001.0g	CC 17/04/97 M. Mc
3	oui	1001.1g	CC 17/04/97 M. Mc
4	oui	1001.0g	CC 17/04/97 M. Mc
5	oui	1001.1g	CC 17/04/97 M. Mc
6	oui	1001.0g	CC 17/04/97 M. Mc

Conditions7° 37°C  $\pm$  0,5

CC 17/04/97 M. Mc

② 75 TPN

CC 17/04/97 M. Mc

3. Pesée des comprimés Bal GAL 205

17.04.97	13:29:38	1	0.0 mg	2	0.0 mg	3	0.0 mg	4	0.0 mg	5	0.0 mg	6	0.0 mg
Code	2403.01-19		695.7 mg		696.3 mg		692.9 mg		698.4 mg		696.8 mg		689.6 mg
ID		ID		ID		ID		ID		ID		ID	

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FOURNIER 1001680

RC 10.07.97

h. lecture. Bal ~~GAL 825~~ <sup>m</sup> Chromometrie GAL 124

LF 178 TER LOT RG 2403/01 19 KG

04-17-1997 16:00

290.0 1 0.0000\_1  
290.0 2 0.0030\_1  
290.0 3 0.0001\_1  
*m*

*m* Lambda No. Valeur\_E

290.0	$T_0$	1	-0.0000_1
290.0		2	-0.0000_1
290.0		3	-0.0007_1
290.0		4	-0.0005_1
290.0		5	-0.0002_1
290.0		6	-0.0003_1
290.0	$T_5$	7	0.2325_1
290.0		8	0.2388_1
290.0		9	0.2330_1
290.0		10	0.2538_1
290.0		11	0.2492_1
290.0		12	0.2442_1
290.0	$T_{10}$	13	0.7679_1
290.0		14	0.7783_1
290.0		15	0.7536_1

290.0	$T_{20}$	31	1.2438_1
290.0		32	1.2742_1
290.0		33	1.2720_1
290.0		34	1.2492_1
290.0		35	1.2690_1
290.0		36	1.2808_1
290.0	$T_{40}$	37	1.2868_1
290.0		38	1.3237_1
290.0		39	1.3167_1
290.0		40	1.2846_1
290.0		41	1.3206_1
290.0		42	1.3015_1
290.0	$T_{50}$	43	1.2937_1
290.0		44	1.3342_1
290.0		45	1.3528_1
290.0		46	1.3151_1

FOURNIER 1001681

Highly Confidential  
Subject to  
Protective Order

h. Lechere. Bal ~~GAL 205~~ Chronometre GAL 124  
M

LF 178 TER LOT RG 2403/01 19 KG

04-17-1997 16:00

290.0 1 0.0000\_1  
290.0 2 0.0030\_1  
290.0 M 3 0.0001\_1

Lambda No. Valeur\_E

290.0	T <sub>0</sub>	1	-0.0000_1	290.0	T <sub>30</sub>	31	1.2438_1
290.0		2	-0.0000_1	290.0		32	1.2742_1
290.0		3	-0.0007_1	290.0		33	1.2720_1
290.0		4	-0.0005_1	290.0		34	1.2492_1
290.0		5	-0.0002_1	290.0		35	1.2690_1
290.0		6	-0.0003_1	290.0		36	1.2808_1
290.0	T <sub>5</sub>	7	0.2325_1	290.0	T <sub>40</sub>	37	1.2868_1
290.0		8	0.2388_1	290.0		38	1.3237_1
290.0		9	0.2330_1	290.0		39	1.3167_1
290.0		10	0.2538_1	290.0		40	1.2846_1
290.0		11	0.2492_1	290.0		41	1.3206_1
290.0		12	0.2442_1	290.0		42	1.3015_1
290.0	T <sub>10</sub>	13	0.7679_1	290.0	T <sub>50</sub>	43	1.2937_1
290.0		14	0.7783_1	290.0		44	1.3342_1
290.0		15	0.7536_1	290.0		45	1.3528_1
290.0		16	0.7897_1	290.0		46	1.3151_1
290.0		17	0.7578_1	290.0		47	1.3306_1
290.0		18	0.7675_1	290.0		48	1.3325_1
290.0	T <sub>15</sub>	19	1.0416_1	290.0	T <sub>60</sub>	49	1.3358_1
290.0		20	1.0690_1	290.0		50	1.3358_1
290.0		21	1.0653_1	290.0		51	1.3548_1
290.0		22	1.0615_1	290.0		52	1.3454_1
290.0		23	1.0469_1	290.0		53	1.3640_1
290.0		24	1.0495_1	290.0		54	1.3572_1
290.0	T <sub>20</sub>	25	1.1622_1	290.0	T <sub>70</sub>	55	1.3671_1
290.0		26	1.1928_1	290.0		56	1.3651_1
290.0		27	1.1898_1	290.0		57	1.4042_1
290.0		28	1.1769_1	290.0	M	58	1.3663_1
290.0		29	1.1631_1	290.0		59	1.4022_1
290.0		30	1.1864_1	290.0		60	1.3746_1

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 17/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER RG 2403/01 à 19 KG  
N° CAHIER : LF 178 TER n°1 p 168  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2403RG01 19 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	695,70 /	696,30 /	692,90 /	698,40 /	696,80 /	689,60 /
quantité de principe actif	160,30	160,44	159,65	160,92	160,55	158,89

Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,233 /	0,239 /	0,233 /	0,254 /	0,249 /	0,244 /
10	1000	0,768 /	0,778 /	0,754 /	0,79 /	0,758 /	0,768 /
15	1000	1,042 /	1,069 /	1,065 /	1,062 /	1,047 /	1,05 /
20	1000	1,162 /	1,193 /	1,19 /	1,177 /	1,163 /	1,186 /
30	1000	1,244 /	1,274 /	1,272 /	1,249 /	1,269 /	1,281 /
40	1000	1,287 /	1,324 /	1,317 /	1,285 /	1,321 /	1,302 /

Fournier 1001682

Highly Confidential  
Subject to  
Protective Order

Re 1007 97

290.0 30 1.1864\_1 290.0 60 1.3746\_1

# DISSOLUTION

m:\commun\glq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 17/04/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2  
TITRE : LF 178 TER RG 2403/01 à 19 KG  
N° CAHIER : LF 178 TER n°1 p 168  
FICHIER : M:\commun\glq\donnbase\LF178ter\dissolution\lot 2403RG01 19 kg  
ELUANT : LSNa 0,025 M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 694,4  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	695,70	696,30	692,90	698,40	696,80	689,60
quantité de principe actif	160,30	160,44	159,65	160,92	160,55	158,89

Témoin 100mg/l 0,900

FOURNIER 1001683

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Protective Order

## SUIVI DE LA DISSOLUTION

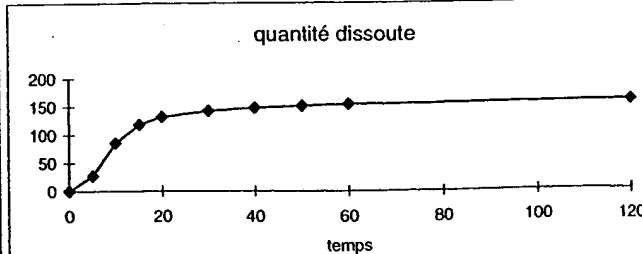
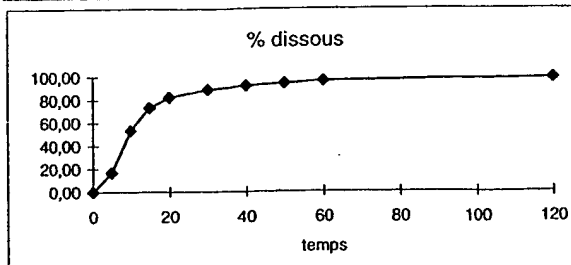
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,233	0,239	0,233	0,254	0,249	0,244
10	1000	0,768	0,778	0,754	0,79	0,758	0,768
15	1000	1,042	1,069	1,065	1,062	1,047	1,05
20	1000	1,162	1,193	1,19	1,177	1,163	1,186
30	1000	1,244	1,274	1,272	1,249	1,269	1,281
40	1000	1,287	1,324	1,317	1,285	1,321	1,302
50	1000	1,294	1,334	1,353	1,315	1,331	1,333
60	1000	1,336	1,336	1,355	1,345	1,364	1,357
120	1000	1,367	1,365	1,404	1,366	1,402	1,375

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	16,79	16,15	16,55	16,22	17,54	17,23	17,06	0,57	3,39
10,0	53,47	53,31	53,96	52,56	54,63	52,54	53,79	0,83	1,55
15,0	73,62	72,57	74,39	74,46	73,69	72,81	73,78	0,79	1,07
20,0	82,50	81,25	83,34	83,53	82,00	81,20	83,66	1,15	1,40
30,0	88,90	87,34	89,37	89,65	87,37	88,93	90,71	1,33	1,50
40,0	92,19	90,75	93,27	93,23	90,29	92,97	92,63	1,32	1,43
50,0	94,08	91,68	94,42	96,19	92,80	94,12	95,25	1,63	1,74
60,0	96,08	95,04	95,02	96,80	95,33	96,87	97,40	1,06	1,10
120,0	98,69	97,65	97,49	100,68	97,24	99,97	99,13	1,44	1,46
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	26,89	25,89	26,56	25,89	28,22	27,67	27,11	0,95	3,55
10,0	85,62	85,46	86,58	83,91	87,92	84,36	85,47	1,47	1,71
15,0	117,88	116,33	119,34	118,88	118,58	116,89	117,23	1,22	1,03
20,0	132,09	130,25	133,71	133,36	131,95	130,36	132,92	1,51	1,14
30,0	142,34	140,00	143,38	143,13	140,60	142,79	144,14	1,65	1,16
40,0	147,62	145,47	149,64	148,84	145,30	149,27	147,18	1,92	1,30
50,0	150,64	146,96	151,49	153,57	149,34	151,12	151,35	2,25	1,49
60,0	153,84	152,35	152,45	154,55	153,41	155,52	154,76	1,30	0,85
120,0	158,03	156,54	156,42	160,74	156,49	160,50	157,51	2,05	1,30
0,0									
0,0									
0,0									





E. 24/5/97

10

Dissolution gélules F173 Ite CANAD

lot 34,

1. Préparation du milieu de dissolution LSWa 0,025M.  
masse molaire du LSWa ~~0,025M~~ 283,4g.

FOURNIER 1001684

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14.05.97

Dissolution de gélules  
de Lipidil Micro 200  
lot 3h Canada

1. Préparation du milieu de dissolution

• eau

On mesure 15 litres à l'aide de la fiole  
jaugée de 5 litres.

Quantité mesurée : 15 litres M 14.05.97 CC 14/05/97

• pesée des LSNa Real M

Pour un milieu à 0,025N, masse de LSNa à  
peser =  $15 \times 0,025 \times 286,4 = 106,2 \text{ g}$ .

Remise à zéro = Tarage M 14.05.97 CC 14/05/97  
gnti pesée = 106,2 g M 14.05.97 CC 14/05/97

LSNa lot: 9616201682

Controle: 0020767 exp 10/12/97

2. Remplissage des bts de dissolutions

1 litre de LSNa 0,025N par bot, mesuré à  
l'aide d'une fiole jaugée

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Protective Order

BC 10.07.97

bol	Quantité mesurée	Vérificateurs
1	1 litre	CC 14/05/97 M 14.05.97
2	1 litre	CC 14/05/97 M 14.05.97
3	1 litre	CC 14/05/97 M 14.05.97
4	1 litre	CC 14/05/97 M 14.05.97
5	1 litre	CC 14/05/97 M 14.05.97
6	1 litre	CC 14/05/97 M 14.05.97

Conditions de dissolution

T° 37°C ± 0,5 AR-JS/5/97 M 15.05.97

③ ~~120 TPN~~ M 15.05.97

③ 75 TPN

AR-JS/5/97 M 15.05.97

Dissolubest DAP : Sotax 6.

3. Pesée des gélules . Bal. GALLEROS

15.05.97 Code ID	07:51:59 Lot 34 lipid micro 200 Canada	1	0.0 mg 421.6 mg
ID		2	0.0 mg 425.0 mg
ID		3	0.0 mg 419.3 mg
ID		4	0.0 mg 426.4 mg
ID		5	0.0 mg 419.8 mg
ID		6	0.1 mg 427.6 mg

*M*

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Protective Order

Rc 10.05.97

h. Lecture

Unikon 942 - chromomètre GAL 122

LIPIDIL MICRO 200 LOT 34 CANADA

05-15-1997 10:57

			Lambda	No.	Valeur_E			
290.0	1	-0.0003_1	290.0	TO 1	0.0013_1	290.0	T <sub>30</sub> 31	1.1402_1
						290.0	T <sub>30</sub> 32	1.1904_1
						290.0	33	1.1616_1
						290.0	34	1.1955_1
						290.0	35	1.1471_1
						290.0	36	1.1794_1
	2	-0.0001_1	290.0	5	0.0020_1	290.0	T <sub>40</sub> 37	1.2239_1
						290.0	T <sub>40</sub> 38	1.2722_1
						290.0	39	1.2423_1
290.0	3	0.0008_1	290.0	6	0.0008_1	290.0	40	1.2778_1
						290.0	41	1.2154_1
						290.0		

FOURNIER 1001687

Highly Confidential  
Subject to  
Protective Order

h. lecture

Uvikon 942 - chromomètre GAL 122

LIPIDIL MICRO 200 LOT 34 CANADA

05-15-1997 10:57

Lambda	No.	Valeur_E
290.0	1	-0.0003_1
290.0	2	-0.0001_1
290.0	3	0.0008_1
290.0	1	0.0013_1
290.0	2	0.0014_1
290.0	3	0.0016_1
290.0	4	0.0015_1
290.0	5	0.0020_1
290.0	6	0.0008_1
290.0	7	0.0507_1
290.0	8	0.1429_1
290.0	9	0.0843_1
290.0	10	0.1877_1
290.0	11	0.0497_1
290.0	12	0.0611_1
290.0	13	0.4905_1
290.0	14	0.6120_1
290.0	15	0.5562_1
290.0	16	0.6362_1
290.0	17	0.4796_1
290.0	18	0.4411_1
290.0	19	0.7941_1
290.0	20	0.8710_1
290.0	21	0.8414_1
290.0	22	0.8767_1
290.0	23	0.7712_1
290.0	24	0.7699_1
290.0	25	0.9724_1
290.0	26	1.0327_1
290.0	27	0.9925_1
290.0	28	1.0213_1
290.0	29	0.9580_1
290.0	30	0.9662_1

290.0	31	1.1402_1
290.0	32	1.1904_1
290.0	33	1.1616_1
290.0	34	1.1955_1
290.0	35	1.1471_1
290.0	36	1.1794_1
290.0	37	1.2239_1
290.0	38	1.2722_1
290.0	39	1.2423_1
290.0	40	1.2778_1
290.0	41	1.2154_1
290.0	42	1.2507_1
290.0	43	1.2946_1
290.0	44	1.3212_1
290.0	45	1.2875_1
290.0	46	1.3510_1
290.0	47	1.2843_1
290.0	48	1.3264_1
290.0	49	1.3061_1
290.0	50	1.3443_1
290.0	51	1.3083_1
290.0	52	1.3498_1
290.0	53	1.3381_1
290.0	54	1.3435_1
290.0	55	1.4522_1
290.0	56	1.5090_1
290.0	57	1.4702_1
290.0	58	1.5024_1
290.0	59	1.4635_1
290.0	60	1.4999_1

## DISSOLUTION

m:\commun\glnq\traitdon\distern5

date édition: le 06/02/97

### CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
DATE : 15/05/97  
APPAREIL : Sotax 6 - Uvikon 942  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lipidil micro 200 Canada lot 34  
N° CAHIER : LI 178ter dissolution n°1 p170  
FICHIER : m:\commun\glnq\donbase\li178ter\dissolution\lot 34 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
200,00	200,00	200,00	200,00	200,00	200,00
masse de la prise d'essai					
quantité de principe actif					

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Témoin 100mg/l 0,900

### SUIVI DE LA DISSOLUTION

TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,051	0,143	0,084	0,188	0,05	0,061
10	1000	0,491	0,612	0,556	0,636	0,48	0,441
15	1000	0,794	0,871	0,841	0,877	0,771	0,77
20	1000	0,972	1,033	0,993	1,021	0,958	0,966
30	1000	1,14	1,19	1,162	1,196	1,147	1,179
40	1000	1,224	1,272	1,242	1,278	1,215	1,251
50	1000	1,295	1,321	1,286	1,351	1,284	1,326

290.0 29 0.9662\_1  
290.0 30 0.9662\_1

290.0 00 1.4999\_1

# DISSOLUTION

m:\commun\l\l\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
DATE : 15/05/97  
APPAREIL : Sotax 6 - Uvikon 942  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lipidil micro 200 Canada lot 34  
N° CAHIER : LI 178ter dissolution n°1 p170  
FICHIER : m:\commun\l\l\donnbase\LI178ter\dissolution\lot 34 Canada  
ELUANT : LNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 200  
dosage théorique 200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001689

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Subject to  
Protective Order

UC 11.07.97

## SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,051	0,143	0,084	0,188	0,05	0,061
10	1000	0,491	0,612	0,556	0,636	0,48	0,441
15	1000	0,794	0,871	0,841	0,877	0,771	0,77
20	1000	0,972	1,033	0,993	1,021	0,958	0,966
30	1000	1,14	1,19	1,162	1,196	1,147	1,179
40	1000	1,224	1,272	1,242	1,278	1,215	1,251
50	1000	1,295	1,321	1,288	1,351	1,284	1,326
60	1000	1,306	1,344	1,308	1,35	1,338	1,344
120	1000	1,452	1,509	1,47	1,502	1,464	1,5

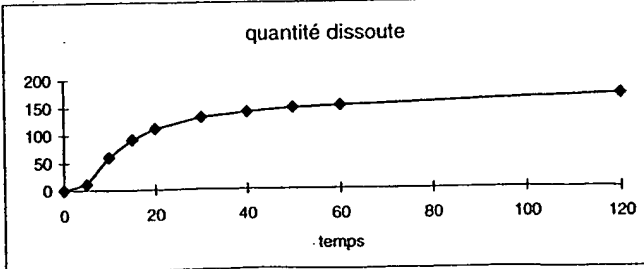
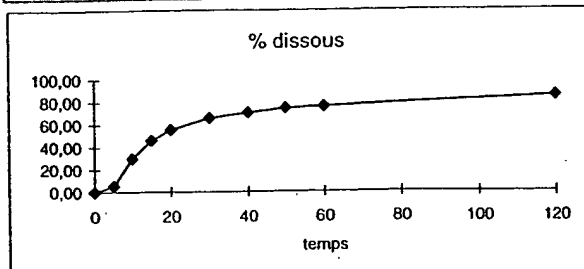
Non retranscrite car différence non significative  
M 11.07.97

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	5,34	2,83	7,94	4,67	10,44	2,78	3,39	3,16	59,16
10,0	29,80	27,29	34,04	30,91	35,39	26,68	24,52	4,34	14,57
15,0	45,77	44,26	48,60	46,90	48,95	42,98	42,92	2,74	5,99
20,0	55,43	54,37	57,84	55,58	57,19	53,58	54,02	1,76	3,17
30,0	65,62	63,97	66,85	65,24	67,20	64,35	66,12	1,32	2,01
40,0	70,28	68,96	71,74	70,01	72,09	68,45	70,45	1,46	2,07
50,0	74,16	73,24	74,81	72,80	76,50	72,62	74,96	1,52	2,06
60,0	75,70	74,21	76,46	74,38	76,82	75,97	76,33	1,12	1,48
120,0	84,46	82,69	86,00	83,74	85,64	83,35	85,37	1,38	1,63
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	10,69	5,67	15,89	9,33	20,89	5,56	6,78	6,32	59,16
10,0	59,61	54,58	68,08	61,82	70,77	53,36	49,03	8,69	14,57
15,0	91,54	88,52	97,20	93,80	97,90	85,96	85,83	5,48	5,99
20,0	110,86	108,74	115,68	111,16	114,39	107,17	108,04	3,52	3,17
30,0	131,25	127,95	133,70	130,49	134,40	128,70	132,24	2,64	2,01
40,0	140,56	137,92	143,47	140,02	144,18	136,89	140,90	2,91	2,07
50,0	148,31	146,48	149,62	145,60	153,00	145,23	149,93	3,05	2,06
60,0	151,39	148,43	152,91	148,76	153,64	151,95	152,66	2,24	1,48
120,0	168,93	165,37	171,99	167,48	171,28	166,69	170,74	2,75	1,63
0,0									
0,0									
0,0									



Le 15 mai 1997

Dissolution de gélules de  
Lipidil Nicot Loc.  
Lot 35 Canada.

1. Préparation du milieu de dissolution. LSNa 0,25M.  
voir p 170.

2. Remplissage des bôles de dissolution.

1 P de LSNa 0,25M mesuré à l'aide d'une fiole jaugée.

bôles	Quantité mesurée	Vérificateurs.
1	1 litre	M 15.05.97 AR-15/5/97
2	1 litre	M 15.05.97 AR-15/5/97
3	1 litre	M 15.05.97 AR-15/5/97
4	1 litre	M 15.05.97 AR-15/5/97
5	1 litre	M 15.05.97 AR-15/5/97
6	1 litre	M 15.05.97 AR-15/5/97

3. Conditions de dissolution

dissolvant DAP 30746.

$T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  M 15.05.97 AR-15/5/97

$\gamma = 75 \text{ rpm}$  M 15.05.97 AR-15/5/97.

4. Pesée des gélules. gal 205.

15.05.97 13:06:09  
Code 35

1	2	3	4	5	6
ID	ID	ID	ID	ID	ID
0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
432.5 mg	426.5 mg	429.6 mg	412.3 mg	420.6 mg	422.0 mg

RC 10.07

05-15-1997 14:20

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	-0.0012_1
290.0	3	-0.0000_1
290.0	4	-0.0000_1
290.0	5	0.0002_1
290.0	6	-0.0000_1
290.0	7	-0.0000_1
290.0	8	-0.0001_1
290.0	9	-0.0001_1
290.0	10	0.0785_1
290.0	11	0.0747_1
290.0	12	0.0532_1
290.0	13	0.0642_1
290.0	14	0.1920_1
290.0	15	0.0653_1
290.0	16	0.4226_1
290.0	17	0.5384_1
290.0	18	0.4532_1
290.0	19	0.4463_1
290.0	20	0.6622_1
290.0	21	0.5188_1
290.0	22	0.7967_1
290.0	23	0.8722_1
290.0	24	0.8309_1
290.0	25	0.7773_1
290.0	26	0.9218_1
290.0	27	0.8446_1
290.0	28	1.0240_1
290.0	29	1.0371_1
290.0	30	1.0277_1
290.0	31	0.9578_1
290.0	32	1.0543_1
290.0	33	1.0198_1
290.0	34	1.2110_1
290.0	35	1.2115_1
290.0	36	1.2150_1
290.0	37	1.1463_1
290.0	38	1.2078_1
290.0	39	1.1692_1
290.0	40	1.3120_1
290.0	41	1.2981_1
290.0	42	1.3130_1
290.0	43	1.2568_1
290.0	44	1.2908_1
290.0	45	1.2902_1
290.0	46	1.3557_1
290.0	47	1.3344_1
290.0	48	1.3350_1
290.0	49	1.3775_1
290.0	50	1.2886_1
290.0	51	1.3333_1
290.0	52	1.4268_1
290.0	53	1.3843_1
290.0	54	1.4026_1
290.0	55	1.3727_1
290.0	56	1.3864_1
290.0	57	1.3744_1

290.0	4	1.4980_1
290.0	5	1.4878_1
290.0	6	1.5001_1
290.0	7	1.4366_1
290.0	8	1.4461_1
290.0	9	1.4557_1

FOURNIER 1001691

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RC 10.07.87



# DISSOLUTION

m:\commun\glnq\traitdon\distem5  
date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 15/05/97  
APPAREIL : Sotax 6 - Uvikon 942  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lipidil micro 200 Canada lot 35  
N° CAHIER : Lf 178ter dissolution n°1 p172  
FICHIER : m:\commun\glnq\donnbase\lf178ter\dissolution\lot 35 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

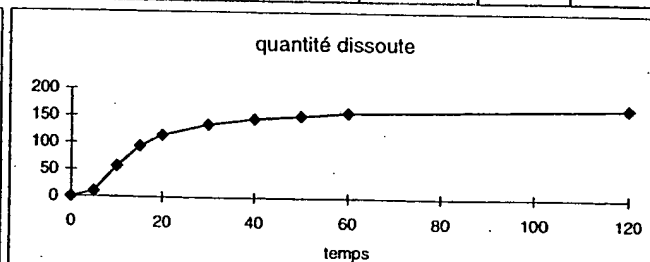
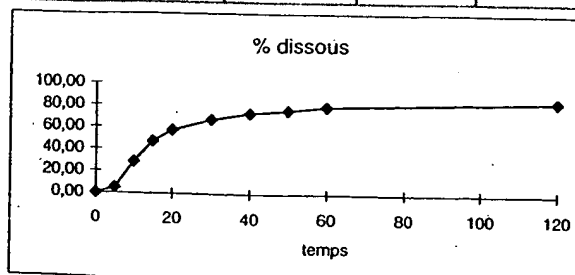
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,079	0,075	0,053	0,064	0,192	0,065
10	1000	0,423	0,538	0,453	0,446	0,662	0,519
15	1000	0,797	0,872	0,831	0,777	0,922	0,845
20	1000	1,024	1,037	1,028	0,958	1,054	1,02
30	1000	1,211	1,212	1,215	1,146	1,208	1,169
40	1000	1,312	1,298	1,313	1,267	1,291	1,29
50	1000	1,356	1,334	1,335	1,378	1,289	1,333
60	1000	1,427	1,384	1,403	1,373	1,386	1,374
120	1000	1,498	1,488	1,5	1,437	1,446	1,456

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	4,89	4,39	4,17	2,94	3,56	10,67	3,61	2,88	58,82
10,0	28,18	23,52	29,91	25,18	24,80	36,83	28,85	4,91	17,41
15,0	46,87	44,42	48,61	46,31	43,31	51,46	47,11	2,94	6,27
20,0	57,07	57,25	58,02	57,48	53,58	59,05	57,06	1,86	3,25
30,0	66,99	67,92	68,03	68,16	64,29	67,90	65,62	1,63	2,43
40,0	72,87	73,87	73,15	73,94	70,78	72,84	72,67	1,15	1,58
50,0	75,68	76,68	75,51	75,53	77,85	73,09	75,42	1,58	2,09
60,0	79,03	81,00	78,66	79,67	77,95	78,84	78,07	1,15	1,45
120,0	83,84	85,34	84,82	85,45	81,89	82,56	83,00	1,55	1,85
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	9,78	8,78	8,33	5,89	7,11	21,33	7,22	5,75	58,82
10,0	56,36	47,04	59,82	50,36	49,59	73,66	57,70	9,82	17,41
15,0	93,74	88,83	97,23	92,61	86,62	102,92	94,21	5,88	6,27
20,0	114,15	114,50	116,05	114,97	107,16	118,10	114,13	3,71	3,25
30,0	133,98	135,85	136,07	136,31	128,58	135,79	131,25	3,26	2,43
40,0	145,75	147,74	146,30	147,88	141,55	145,69	145,34	2,31	1,58
50,0	151,36	153,36	151,02	151,05	155,69	146,18	150,84	3,16	2,09
60,0	158,06	162,00	157,31	159,35	155,90	157,68	156,13	2,29	1,45
120,0	167,69	170,68	169,64	170,91	163,78	165,11	166,01	3,09	1,85
0,0									
0,0									
0,0									



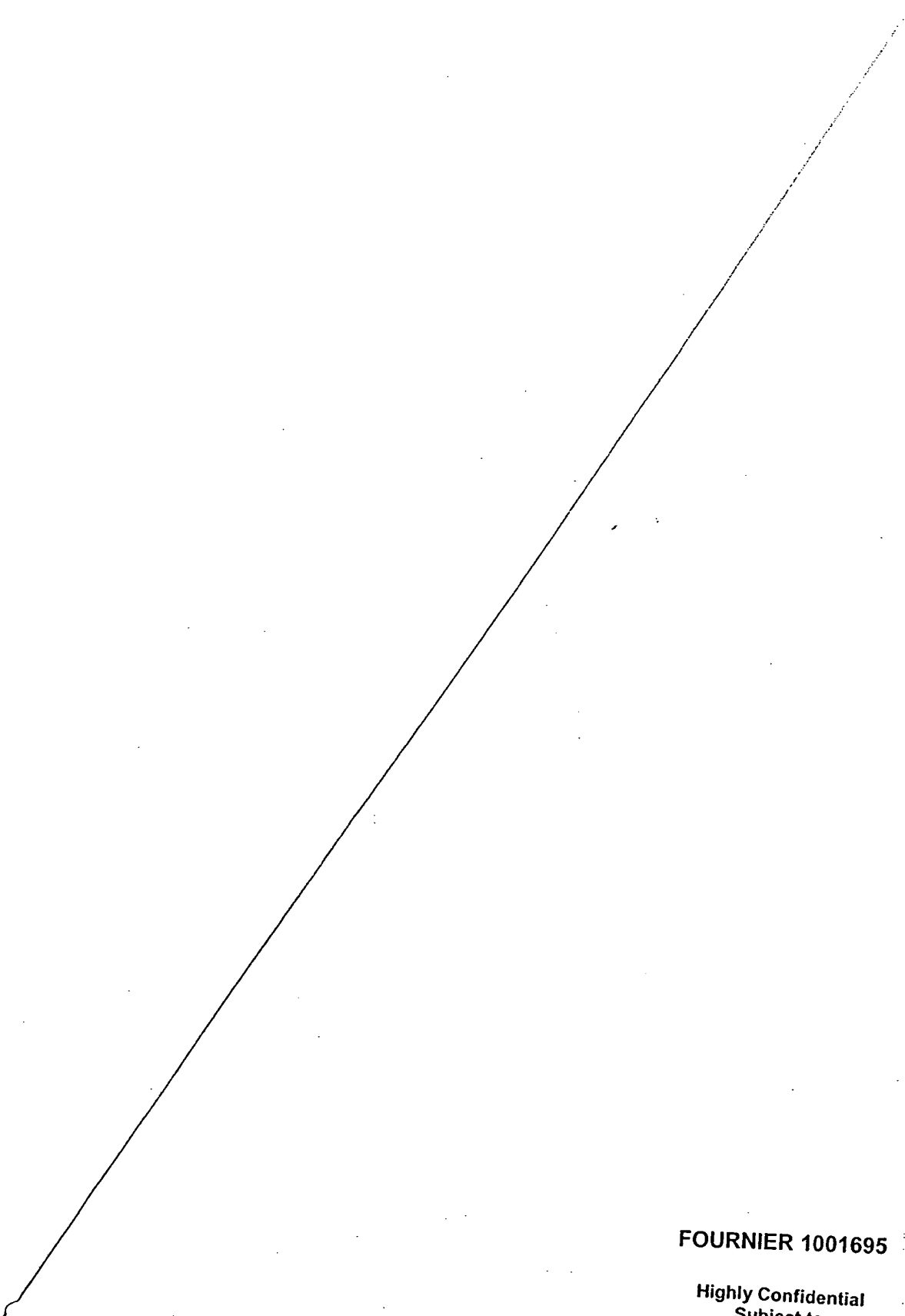
Pl. 10.07.97

RC 10 07 92 - Cloture

FOURNIER 1001693  
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Protective Order

FOURNIER 1001694

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Subject to  
Protective Order



FOURNIER 1001695

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Subject to  
Protective Order



11	08.04.97	Dosage fenofibrate dans MF 2394/01 RG et 2396/01 RG
116	09.04.97	Dissolution - 2397/01 RG - 14 Kg
118	10.04.97	Dissolution - 2397/01 RG - 18 Kg
121	10.04.97	Dissolution 2398/01 RG - 14 Kg
124	10.04.97	Dissolution 2398/01 RG - 18 Kg
126	10.04.97	Dosage du fenofibrate dans MF - 2397/01 RG, 2398/01 RG
130	11.04.97	Dissolution - 2399/01 RG - 15 Kg
132	11.04.97	Dosage fenofibrate dans MF - 2399/01 RG
135	11.04.97	Dissolution - 2399/01 RG - 20 Kg -
138	14.04.97	Dissolution - 2400/01 RG - 14 Kg
141	14.04.97	Dissolution - 2400/01 RG - 18 Kg
143	15.04.97	Dissolution - 2401/01 RG - 15 Kg
146	15.04.97	Dissolution - lipide P Micro - lot 51
149	15.04.97	Dissolution - 2401/01 RG - 20 Kg
151	16.04.97	Dissolution - 2402/01 RG - 15 Kg
154	16.04.97	Dissolution - lipide P Micro - lot 51 -
156	16.04.97	Dissolution - 2402/01 RG - 20 Kg
158	16.04.97	Dissolution - lipide P Micro - lot 52 -
161	17.04.97	Dissolution - 2403/01 RG - 15 Kg
164	17.04.97	Dissolution - lipide P Micro - lot 53
167	17.04.97	Dissolution - 2403/02 RG - 19 Kg
170	14.05.97	Dissolution - lipide P Micro - lot 34 -
173	15.05.97	Dissolution - lipide P Micro - lot 35 -

RG 10.07.97 Cloture

FOURNIER 1001696

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page	Date	Dénomination
103	18.02.97	Essais dissolution Tween 80
5 à 21	19/02/97 ou 24/02/97	Préparations de solutions de fénofibrate Réalisation de spectres d'absorption pour gamme étalon
23	26/02/97	Préparation d'une solution de fénofibrate dans l'éthanol → spectre d'absorption.
27	26/02/97	Dissolution dans tween 80 27. 75 TPN. lot 340.
31	27/02/97	Dissolution dans tween 80 27. 75 TPN. gélules Lip 200 ARR 1710.
36	26.02.97	Dissolution - cpr lot 340 - $LSNa\ 0,02M$ - 75 rpm.
140	26.02.97	Dissolution - gélules lip 200 - an 1710 - $LSNa\ 0,02M$ - 75 rpm.
143	28.02.97	Dissolution - cpr lot 340 - $LSNa\ 0,025M$ - 75 rpm.
147	04.03.97	Dissolution - gélules lip 200 - an 1710 - $LSNa\ 0,025M$ - 75 rpm.
152	06.03.97	Concentration à saturation du cominonival dans $LSNa\ 0,025M$ .
155	10.03.97	Dissolution lot 351 - $LSNa\ 0,025M$ - 75 rpm.
158	11.03.97	Dissolution lot 354 - $LSNa\ 0,025M$ - 75 rpm.
061	11.03.97	Dissolution lot 358 - $LSNa\ 0,025M$ - 75 rpm.
164	13.03.97	Dissolution lot 361 - $LSNa\ 0,025M$ - 75 rpm.
168	14.03.97	Dissolution lot 334 - $LSNa\ 0,025M$ - 75 rpm.
172	20.03.97	Dissolution Lipidil 200 - lot 48 - 49 - $LSNa\ 0,025M$ - 75 rpm.
181	25.03.97	Dissolution Lipanthyl M - lot 2177 " "
185	03.04.97	Dissolutions CF/18TER lot 2394/01 RG - 14 et 18 Kg - " "
192	07.04.97	Dissolution 2393/01 RG - opadry OYR.
195	07.04.97	Dissolution - 2394/01 RG - 14 Kg
197	07.04.97	Dissolution - 2394/01 RG - 18 Kg
199	07.04.97	Dosage fénofibrate lot 2394/01 RG - 14 Kg et 18 Kg
104	08.04.97	Dissolution - lot 2396/01 RG - 18 Kg
106	08.04.97	Dissolution 2393/01 RG enrobés opadry - 75 jours à 75% RH
109	08.04.97	Dissolution - lot 2396/01 RG 14 Kg

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Je SOUSIGNÉ Maître Patrick NOURISSAT, notaire associé  
de la Société Civile Professionnelle " Patrick NOURISSAT, Didier  
NOURISSAT et Hugues MISSEREY " titulaire d'un office Notarial  
dont le siège est à DUON, 25 rue Buffon.

CERTIFIE ET ATTESTE avoir procédé ce jour à la  
clôture du présent registre contenant 12 pages,  
l'avoir coté et paraphé en dernière page, destiné à  
l'enregistrement des formulations de médicaments pour  
les Laboratoires FOURNIER, centre de Biogalénique,  
42 rue de Longvic à Chenôve.

FAIT POUR SERVIR ET VALOIR CE QUE DE DROIT  
A DUON

L'an DEUX MILLE DIX

LE 26 Juin



FOURNIER 1001698

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Subject to  
Protective Order

Cahier LF 178 ter  
Dissolution n°2

FOURNIER 1001699

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Protective Order



01

CAHIER LF 178 Ter

DISSOLUTION N° 2

Le cahier contient 180 pages et comporte une erreur  
de pagination à la page 152 qui est doublée  
DT 16/6/97.

Cahier approuvé par DT le 26/04/00  
DT 26/04/00

FOURNIER 1001700

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Protective Order

Commencé le 15 Nov 97 - Terminé le 30.07.97.

Dissolution de gélules  
de Lipidil Niers 200

lot 37 Canada

1. Préparation du milieu de dissolution

• Eau

On mesure 28 litres à l'aide de fioles jaugées  
5 et 1 litres.

Quantité mesurée : 28 litres M 15.05.97  
CC 15/05/97

• Pesée du LiNa. Bal GAL III

Pour un milieu à 0,025 N, masse de LiNa à  
peser =  $28 \times 0,025 \times 286,4 = 201,88 \text{ g}$ .

- Tarage du Bécher M 15.05.97 AL-1515/97

- Quantité pesée : 201,9 g. M 15.05.97 15/05/97

LiNa lot 961620/1682 - n° Clé : 0020767.

2. Remplissage des bds de dissolution

1 litre de LiNa 0,025 N par bd, mesurer à l'aide  
d'une fiole jaugée

FOURNIER 1001701

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PL 24.06.97

bd	Quantité mesurée	Vérificateurs
1	1 litre	cc 15/05/97 M. B. O. S. 97
2	1 litre	cc 15/05/97 M. 15.05.97
3	1 litre	cc 15/05/97 M. 15.05.97
4	1 litre	cc 15/05/97 M. 15.05.97
5	1 litre	cc 15/05/97 M. 15.05.97
6	1 litre	cc 15/05/97 M. 15.05.97

Condition de  
dissolution

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5$  AG 16/15/97 M. 16.05.97

$\phi = 75$  TPN AG 16/15/97. M. 16.05.97

Dissolutest DAP: Solax n°5

3. Pesée des gélules Bal GAL 205

15.05.97 Code Lot 37 Lipid.1 micro bio Canada	1	2	3	4	5	6
ID	ID	ID	ID	ID	ID	ID
0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
433.3 mg	426.5 mg	430.8 mg	426.1 mg	429.3 mg	423.2 mg	

4. Lechete

Unikon Bio-DAP - Chronomètre GAL 122

FOURNIER 1001702

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Protective Order

PC 24.06.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
DATE : 16/05/97  
APPAREIL : Sotax 5 - Uvikon 810  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lipidil micro 200 Canada lot 37  
N° CAHIER : LI 178ter dissolution n°2 p 2  
FICHIER : m:\commun\glnq\donnbase\LI178ter\dissolution\lot 37 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001703

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## SUIVI DE LA DISSOLUTION

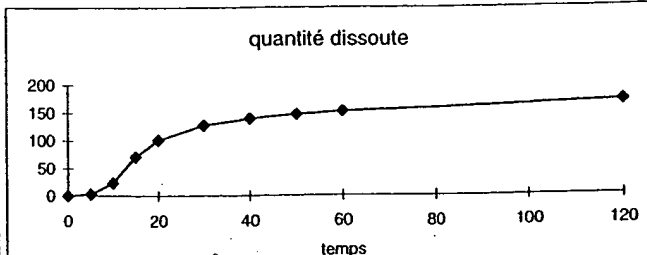
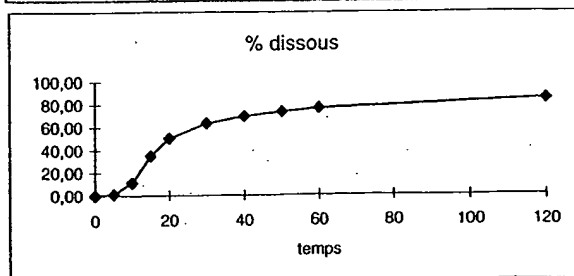
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,01	0,018	0,009	0,023	0,063	0,017
10	1000	0,207	0,211	0,161	0,259	0,236	0,152
15	1000	0,648	0,657	0,619	0,703	0,633	0,522
20	1000	0,92	0,902	0,902	0,944	0,93	0,837
30	1000	1,156	1,12	1,127	1,163	1,132	1,096
40	1000	1,26	1,246	1,226	1,245	1,248	1,206
50	1000	1,34	1,292	1,282	1,299	1,311	1,273
60	1000	1,382	1,34	1,342	1,347	1,335	1,318
120	1000	1,538	1,478	1,502	1,496	1,497	1,453

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	1,30	0,56	1,00	0,50	1,28	3,50	0,94	1,12	86,26
10,0	11,36	11,50	11,73	8,95	14,40	13,13	8,45	2,31	20,38
15,0	35,08	36,06	36,56	34,44	39,13	35,25	29,05	3,36	9,58
20,0	50,56	51,35	50,36	50,33	52,72	51,93	46,69	2,11	4,17
30,0	63,40	64,72	62,72	63,08	65,15	63,41	61,31	1,39	2,20
40,0	69,61	70,82	70,03	68,89	70,03	70,17	67,73	1,11	1,60
50,0	73,34	75,61	72,93	72,35	73,37	74,01	71,79	1,36	1,85
60,0	76,18	78,32	75,96	76,04	76,40	75,71	74,64	1,21	1,58
120,0	84,88	87,37	84,00	85,30	85,05	85,08	82,51	1,60	1,89
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	2,59	1,11	2,00	1,00	2,56	7,00	1,89	2,24	86,26
10,0	22,72	23,01	23,45	17,89	28,79	26,26	16,90	4,63	20,38
15,0	70,16	72,12	73,13	68,87	78,27	70,50	58,09	6,72	9,58
20,0	101,12	102,70	100,71	100,66	105,44	103,85	93,38	4,21	4,17
30,0	126,79	129,44	125,44	126,16	130,29	126,81	122,63	2,79	2,20
40,0	139,22	141,63	140,06	137,79	140,05	140,33	135,46	2,22	1,60
50,0	146,69	151,22	145,86	144,69	146,74	148,02	143,57	2,71	1,85
60,0	152,35	156,63	151,91	152,07	152,80	151,42	149,28	2,41	1,58
120,0	169,77	174,74	167,99	170,59	170,10	170,16	165,01	3,21	1,89
0,0									
0,0									
0,0									



ML

RC09.07.97

RC.24.06.97

1 .000 ABS 290.0 NM  
 1 -.000 ABS 290.0 NM  
 1 .000 ABS 290.0 NM

1 *T5* .000 ABS 290.0 NM  
 1 .002 ABS 290.0 NM  
 1 .003 ABS 290.0 NM  
 1 .002 ABS 290.0 NM  
 1 .002 ABS 290.0 NM  
 1 .002 ABS 290.0 NM

1 *T5* .010 ABS 290.0 NM  
 1 .018 ABS 290.0 NM  
 1 .000 ABS 290.0 NM  
 1 .020 ABS 290.0 NM  
 1 .000 ABS 290.0 NM  
 1 .010 ABS 290.0 NM

1 *T10* .200 ABS 290.0 NM  
 1 .211 ABS 290.0 NM  
 1 .151 ABS 290.0 NM  
 1 .259 ABS 290.0 NM  
 1 .236 ABS 290.0 NM  
 1 .152 ABS 290.0 NM

1 *T15* .648 ABS 290.0 NM  
 1 .657 ABS 290.0 NM  
 1 .619 ABS 290.0 NM  
 1 .700 ABS 290.0 NM  
 1 .633 ABS 290.0 NM  
 1 .522 ABS 290.0 NM

1 *T20* .920 ABS 290.0 NM  
 1 .982 ABS 290.0 NM  
 1 .902 ABS 290.0 NM  
 1 .900 ABS 290.0 NM  
 1 .900 ABS 290.0 NM  
 1 .900 ABS 290.0 NM

1 *T30* 1.100 ABS 290.0 NM  
 1 1.100 ABS 290.0 NM  
 1 1.100 ABS 290.0 NM  
 1 1.100 ABS 290.0 NM  
 1 1.132 ABS 290.0 NM  
 1 1.096 ABS 290.0 NM

1 *T40* 1.260 ABS 290.0 NM  
 1 1.260 ABS 290.0 NM  
 1 . ABS 290.0 NM  
 1 . ABS 290.0 NM  
 1 . ABS 290.0 NM  
 1 1.266 ABS 290.0 NM

1 *T50* 1.340 ABS 290.0 NM  
 1 1.292 ABS 290.0 NM  
 1 1.282 ABS 290.0 NM  
 1 1.299 ABS 290.0 NM  
 1 1.311 ABS 290.0 NM  
 1 1.273 ABS 290.0 NM

1 *T60* 1.340 ABS 290.0 NM  
 1 1.340 ABS 290.0 NM  
 1 . ABS 290.0 NM  
 1 . ABS 290.0 NM  
 1 . ABS 290.0 NM  
 1 . ABS 290.0 NM

1 *T60* 1.400 ABS 290.0 NM  
 1 1.400 ABS 290.0 NM  
 1 1.382 ABS 290.0 NM  
 1 1.400 ABS 290.0 NM  
 1 1.497 ABS 290.0 NM  
 1 1.400 ABS 290.0 NM

CV
86,26
20,38
9,58
4,17
2,20
1,60
1,85
1,58
1,89

CV
86,26
20,38
9,58
4,17
2,20
1,60
1,85
1,58
1,89

FOURNIER 1001704

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# DISSOLUTION

m:\commun\gl\ql\traitdon\distem5

date édition: le 06/02/97

005

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
DATE : 16/05/97  
APPAREIL : Solax 5 - Uvikon 810  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lipidil micro 200 Canada lot 37  
N° CAHIER : LI 178ter dissolution n°2 p. 502 RC  
FICHIER : m:\commun\gl\ql\donnbase\LI 178ter\dissolution\lot 37 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 200  
dosage théorique 200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001705

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## SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,01	0,018	0,009	0,023	0,063	0,017
10	1000	0,207	0,211	0,161	0,259	0,236	0,152
15	1000	0,648	0,657	0,619	0,703	0,633	0,522
20	1000	0,92	0,902	0,902	0,944	0,93	0,837
30	1000	1,156	1,12	1,127	1,163	1,132	1,096
40	1000	1,236	1,246	1,226	1,245	1,248	1,206
50	1000	1,34	1,292	1,282	1,299	1,311	1,273
60	1000	1,382	1,34	1,342	1,347	1,335	1,318
120	1000	1,538	1,478	1,502	1,496	1,497	1,453

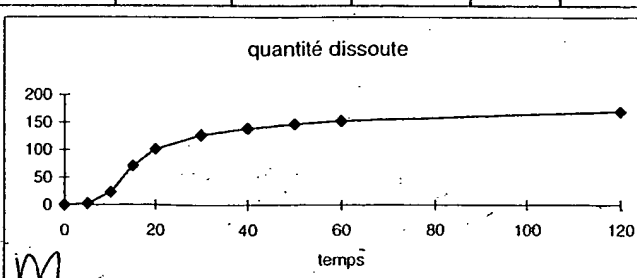
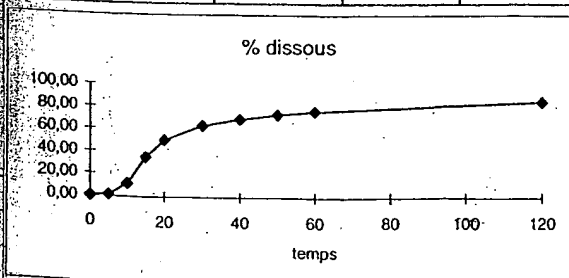
Faute de calcul réhaite avec la  
valeur correcte M 06.07.97

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	1,30	0,56	1,00	0,50	1,28	3,50	0,94	1,12	86,26
10,0	11,36	11,50	11,73	8,95	14,40	13,13	8,45	2,31	20,38
15,0	35,08	36,06	36,56	34,44	39,13	35,25	29,05	3,36	9,58
20,0	50,56	51,35	50,36	50,33	52,72	51,93	46,69	2,11	4,17
30,0	63,40	64,72	62,72	63,08	65,15	63,41	61,31	1,39	2,20
40,0	69,30	68,93	70,03	68,89	70,03	70,17	67,73	0,96	1,38
50,0	73,34	75,60	72,93	72,35	73,37	74,01	71,79	1,35	1,84
60,0	76,17	78,31	75,96	76,04	76,40	75,71	74,64	1,20	1,58
120,0	84,88	87,36	84,00	85,30	85,05	85,08	82,51	1,60	1,89
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	2,59	1,11	2,00	1,00	2,56	7,00	1,89	2,24	86,26
10,0	22,72	23,01	23,45	17,89	28,79	26,26	16,90	4,63	20,38
15,0	70,16	72,12	73,13	68,87	78,27	70,50	58,09	6,72	9,58
20,0	101,12	102,70	100,71	100,66	105,44	103,85	93,38	4,21	4,17
30,0	126,79	129,44	125,44	126,16	130,29	126,81	122,63	2,79	2,20
40,0	138,59	137,86	140,06	137,79	140,05	140,33	135,46	1,91	1,38
50,0	146,68	151,20	145,86	144,69	146,74	148,02	143,57	2,70	1,84
60,0	152,35	156,62	151,91	152,07	152,80	151,42	149,28	2,41	1,58
120,0	169,76	174,72	167,99	170,59	170,10	170,16	165,01	3,20	1,89
0,0									
0,0									
0,0									



Le 16/5/97

Dissolution gélules lipidiP-80H  
lot 38 Canada.

1. Préparation du LSNa 0,025H

voir proc.

FOURNIER 1001706

Highly Confidential  
Subject to  
Protective Order2. Remplissage des bords de dissolution.

1L de LSNa 0,025H mesurée à l'éprouvette graduée de 1L.

bords	volume mesuré	Signature.
1	1 litre	M 15.05.97 AB 15/5/97
2	1 litre	M 15.05.97 AB 15/5/97
3	1 litre	M 15.05.97 AB 15/5/97
4	1 litre	M 15.05.97 AB 15/5/97
5	1 litre	M 15.05.97 AB 15/5/97
6	1 litre	M 15.05.97 AB 15/5/97

3. Conditions de dissolution.

dissolution dans AT7 Dap 106.

 $T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  M 16.05.97 AB 16/5/97 $\sigma = 75 \text{ rpm}$  M 16.05.97 AB 16/5/974. Pesée des gélules gélules.

AL 24.06.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## DITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 16/05/97  
APPAREIL : Sotax 6 - Uvikon 942  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lipidil micro 200 Canada lot 38  
N° CAHIER : LI 178ter dissolution n°2 p6  
FICHER : m:\commun\glnq\donnbase\li178ter\dissolution\lot 38 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	200,00	200,00	200,00	200,00	200,00	200,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001707

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Subject to  
Protective Order

## SUIVI DE LA DISSOLUTION

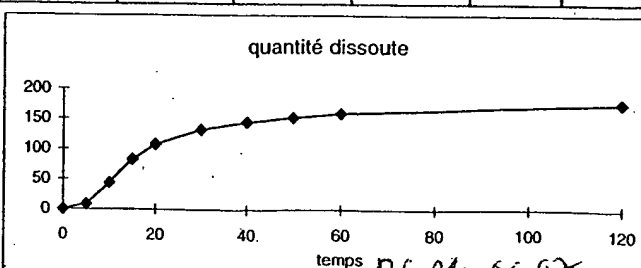
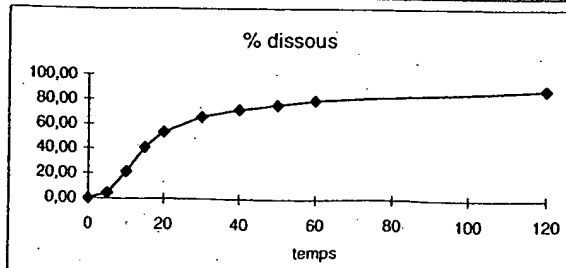
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,063	0,076	0,099	0,084	0,04	0,114
10	1000	0,329	0,368	0,485	0,27	0,325	0,569
15	1000	0,722	0,745	0,775	0,678	0,664	0,853
20	1000	0,965	0,944	0,964	0,898	0,947	1,025
30	1000	1,159	1,187	1,177	1,139	1,185	1,215
40	1000	1,273	1,293	1,279	1,26	1,294	1,289
50	1000	1,335	1,352	1,328	1,316	1,375	1,372
60	1000	1,384	1,403	1,389	1,384	1,419	1,418
120	1000	1,523	1,551	1,531	1,526	1,581	1,562

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	4,41	3,50	4,22	5,50	4,67	2,22	6,33	1,46	33,04
10,0	21,74	18,30	20,47	26,97	15,02	18,07	31,64	6,29	28,91
15,0	41,21	40,22	41,51	43,22	37,77	36,99	47,58	3,88	9,42
20,0	53,51	53,92	52,77	53,93	50,18	52,90	57,37	2,34	4,37
30,0	65,99	64,97	66,54	66,03	63,81	66,38	68,21	1,49	2,27
40,0	72,11	71,62	72,76	72,03	70,85	72,77	72,66	0,77	1,07
50,0	76,08	75,42	76,39	75,11	74,31	77,63	77,63	1,37	1,80
60,0	79,41	78,51	79,60	78,86	78,46	80,45	80,57	0,95	1,19
120,0	87,92	86,62	88,21	87,14	86,73	89,85	88,96	1,31	1,49
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	8,81	7,00	8,44	11,00	9,33	4,44	12,67	2,91	33,04
10,0	43,49	36,59	40,93	53,94	30,05	36,13	63,29	12,57	28,91
15,0	82,43	80,44	83,02	86,44	75,53	73,98	95,16	7,76	9,42
20,0	107,02	107,84	105,55	107,87	100,35	105,79	114,74	4,67	4,37
30,0	131,98	129,93	133,07	132,07	127,63	132,76	136,42	2,99	2,27
40,0	144,23	143,24	145,51	144,06	141,71	145,53	145,32	1,54	1,07
50,0	152,16	150,84	152,79	150,21	148,63	155,25	155,26	2,74	1,80
60,0	158,82	157,03	159,20	157,73	156,91	160,91	161,13	1,89	1,19
120,0	175,84	173,24	176,43	174,28	173,46	179,69	177,92	2,62	1,49
0,0									
0,0									
0,0									



RC 24.06.97



16.05.97 08:26:47

38

Code

ID

0.0000 g

0.4285 g

0.0000 g

0.4210 g

0.0000 g

0.4248 g

0.0000 g

0.4225 g

0.0000 g

0.4287 g

0.0000 g

0.4282 g

ID

ID

ID

ID

ID

S. Lorne

sur spectrophotomètre DAP KONTROL 242

cuvettes de 2 mm de trajet optique.

FOURNIER 1001708

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Protective Order

GELULES LIPIDIL 200M CANADA LOT 38

05-16-1997 10:38

Lambda No. Valeur\_E

290.0	1	0.0000_1
290.0	2	-0.0002_1
290.0	3	0.0000_1
290.0	4	0.0001_1
290.0	5	0.0001_1
290.0	6	0.0000_1
290.0	7	0.0000_1
290.0	8	-0.0000_1
290.0	9	0.0000_1
290.0	10	0.0630_1
290.0	11	0.0763_1
290.0	12	0.0994_1
290.0	13	0.0836_1
290.0	14	0.0395_1
290.0	15	0.1143_1
290.0	16	0.3290_1
290.0	17	0.3676_1
290.0	18	0.4851_1
290.0	19	0.2697_1
290.0	20	0.3250_1
290.0	21	0.5693_1
290.0	22	0.7220_1
290.0	23	0.7448_1
290.0	24	0.7750_1
290.0	25	0.6781_1
290.0	26	0.6636_1
290.0	27	0.8534_1
290.0	28	0.9649_1
290.0	29	0.9440_1
290.0	30	0.9640_1
290.0	31	0.8980_1
290.0	32	0.9474_1
290.0	33	1.0250_1

290.0	34	1.1589_1
290.0	35	1.1871_1
290.0	36	1.1767_1
290.0	37	1.1392_1
290.0	38	1.1848_1
290.0	39	1.2146_1
290.0	40	1.2733_1
290.0	41	1.2928_1
290.0	42	1.2793_1
290.0	43	1.2597_1
290.0	44	1.2937_1
290.0	45	1.2894_1
290.0	46	1.3348_1
290.0	47	1.3515_1
290.0	48	1.3275_1
290.0	49	1.3164_1
290.0	50	1.3753_1
290.0	51	1.3715_1
290.0	52	1.3841_1
290.0	53	1.4025_1
290.0	54	1.3887_1
290.0	55	1.3841_1
290.0	56	1.4192_1
290.0	57	1.4179_1
290.0	58	1.5228_1
290.0	59	1.5507_1
290.0	60	1.5308_1
290.0	61	1.5257_1
290.0	62	1.5813_1
290.0	63	1.5624_1

L 16/5/97.

Disbolition gélules Apidip bon  
 lot 39 Canada.

### 1. Préparation du milieu de dissolution

LSNa 0,25 M ou 0,02

### 2. Mesure du volume dans les bols

1 litre de LSNa 0,25 M mesuré à l'éprouvette graduée.

bol	volume mesuré	Signature.
1	1 l.	SL 16/05/97. AL 16/5/97
2	1 l	SL 16/05/97 AL 16/5/97
3	1 l	SL 16/05/97 AL 16/5/97
4	1 l	SL 16/05/97 AL 16/5/97
5	1 l	SL 16/05/97 AL 16/5/97
6	1 l	SL 16/05/97 AL 16/5/97

### 3. Pesée des gélules

gél. 39

16.05.97 12:41:10  
 Code 39

0.0 mg

423.1 mg

ID

0.0 mg

423.3 mg

ID

0.0 mg

423.6 mg

ID

0.0 mg

425.5 mg

ID

0.0 mg

423.3 mg

ID

0.0 mg

419.7 mg

FOURNIER 1001709  
 Highly Confidential  
 Subject to  
 Protective Order

Recl 06.97

## 4. Conditions opératoires

dissolvant: SMAX AT7 ~~DAE~~ DAP no 6 $T^0 = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  AG 16/5/97 M 16.05.97

S. 75 TPN. AP 16/5/97 M 16.05.97

## 5. Lecture

sur spectrophotométrie KONTON 912 DAP

GELULES LIPIDIL 200M CANADA LOT 39

05-16-1997 13:48

Lambda No. Valeur\_E

290.0	1	-0.0002_1
290.0	2	0.0028_1
290.0	3	0.0000_1
290.0	4	-0.0029_1
290.0	5	-0.0000_1
290.0	6	-0.0000_1
290.0	7	-0.0001_1
290.0	8	-0.0001_1
290.0	9	-0.0001_1
290.0	10	0.0426_1
290.0	11	0.0962_1
290.0	12	0.0879_1
290.0	13	0.0548_1
290.0	14	0.0939_1
290.0	15	0.1159_1
290.0	16	0.2733_1
290.0	17	0.4915_1
290.0	18	0.3330_1
290.0	19	0.4269_1
290.0	20	0.4489_1
290.0	21	0.4482_1
290.0	22	0.7058_1
290.0	23	0.8232_1
290.0	24	0.7269_1
290.0	25	0.7846_1
290.0	26	0.8071_1
290.0	27	0.8010_1
290.0	28	0.9452_1
290.0	29	1.0164_1
290.0	30	0.9693_1
290.0	31	1.0009_1
290.0	32	1.0011_1
290.0	33	0.9988_1

290.0	34	1.1785_1
290.0	35	1.2216_1
290.0	36	1.1795_1
290.0	37	1.2032_1
290.0	38	1.1792_1
290.0	39	1.1841_1
290.0	40	1.2761_1
290.0	41	1.3070_1
290.0	42	1.2899_1
290.0	43	1.2767_1
290.0	44	1.2864_1
290.0	45	1.2966_1
290.0	46	1.3519_1
290.0	47	1.3798_1
290.0	48	1.3730_1
290.0	49	1.3794_1
290.0	50	1.3631_1
290.0	51	1.3695_1
290.0	52	1.4047_1
290.0	53	1.4224_1
290.0	54	1.4160_1
290.0	55	1.4131_1
290.0	56	1.4070_1
290.0	57	1.4240_1
290.0	58	1.5379_1
290.0	59	1.5424_1
290.0	60	1.5691_1
290.0	61	1.5461_1
290.0	62	1.5125_1
290.0	63	1.5628_1

FOURNIER 1001710

Highly Confidential  
Subject to  
Protective Order

RC 24.06.97

# DISSOLUTION

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 16/05/97  
APPAREIL : Sotax 6 - Uvikon 942  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : gélules lipidil micro 200 Canada lot 39  
N° CAHIER : U 178ter dissolution n°2 p8  
FICHIER : m:\commun\glnq\donnbase\U178ter\dissolution\lot 39 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

m:\commun\glnq\traitdon\dstem5  
date édition: le 06/02/97

## PREPARATION DES ECHANTILLONS

masse théorique	200
dosage théorique	200 en mg

masse de la prise d'essai	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00
	200,00	200,00	200,00	200,00	200,00	200,00

**FOURNIER 1001711**  
Highly Confidential  
Subject to  
Protective Order

## SUIVI DE LA DISSOLUTION

Témoin 100mg/l 0,900

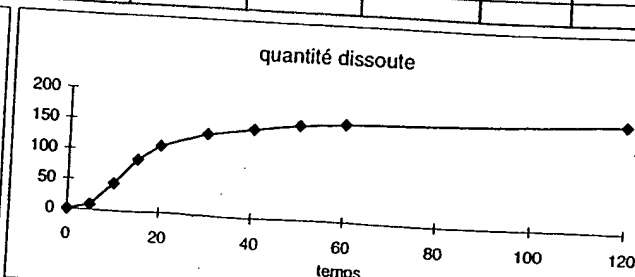
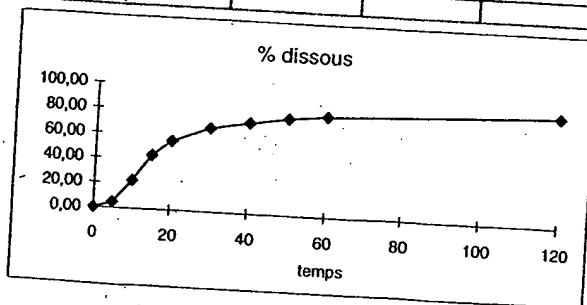
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,043	0,096	0,088	0,055	0,094	0,116
10	1000	0,273	0,492	0,333	0,427	0,449	0,448
15	1000	0,706	0,823	0,727	0,785	0,807	0,801
20	1000	0,945	1,016	0,969	1,001	1,001	0,999
30	1000	1,179	1,222	1,18	1,203	1,179	1,184
40	1000	1,276	1,307	1,29	1,277	1,286	1,297
50	1000	1,352	1,38	1,373	1,379	1,363	1,37
60	1000	1,405	1,422	1,416	1,413	1,407	1,424
120	1000	1,538	1,542	1,569	1,546	1,513	1,563

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	4,56	2,39	5,33	4,89	3,06	5,22	6,44	1,53	33,54
10,0	22,45	15,18	27,36	18,52	23,74	24,97	24,92	4,62	20,56
15,0	43,18	39,31	45,89	40,51	43,75	44,98	44,66	2,65	6,14
20,0	55,27	52,78	56,84	54,15	55,96	55,99	55,88	1,50	2,71
30,0	66,80	66,05	68,56	66,14	67,46	66,15	66,43	1,01	1,51
40,0	72,56	71,76	73,62	72,58	71,91	72,43	73,04	0,70	0,96
50,0	77,40	76,34	78,04	77,55	77,93	77,06	77,46	0,63	0,81
60,0	80,28	79,66	80,76	80,32	80,20	79,88	80,84	0,47	0,58
120,0	87,93	87,44	87,82	89,22	87,98	86,16	88,96	1,10	1,25
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	9,11	4,78	10,67	9,78	6,11	10,44	12,89	3,06	33,54
10,0	44,90	30,36	54,72	37,05	47,48	49,94	49,84	9,23	20,56
15,0	86,36	78,62	91,77	81,01	87,49	89,97	89,31	5,31	6,14
20,0	110,53	105,57	113,67	108,30	111,93	111,97	111,76	3,00	2,71
30,0	133,60	132,09	137,13	132,29	134,93	132,31	132,87	2,02	1,51
40,0	145,11	143,53	147,25	145,17	143,82	144,85	146,08	1,40	0,96
50,0	154,79	152,68	156,09	155,10	155,86	154,12	154,91	1,25	0,81
60,0	160,55	159,32	161,52	160,64	160,40	159,77	161,68	0,93	0,58
120,0	175,86	174,88	175,64	178,43	175,97	172,33	177,91	2,21	1,25
0,0									
0,0									
0,0									



RC24.06.97

16.05.97

# Dissolution de gélules de Lipidil Micro 200

011

lot 40 Canada

## 1. Préparation du milieu de dissolution

Voir page 2

## 2. Remplissage des bts de dissolution

1 litre de l3Na 0,025N par bt, mesuré à l'aide  
d'une fiole jaugée de 1 litre.

bt	quantité mesurée	Vérificateurs
1	1 litre	M 16.05.97 VA 16.05.97
2	1 litre	M 16.05.97 VA 16.05.97
3	1 litre	M 16.05.97 VA 16.05.97
4	1 litre	M 16.05.97 VA 16.05.97
5	1 litre	M 16.05.97 VA 16.05.97
6	1 litre	M 16.05.97 VA 16.05.97

Conditions dedissolution

T° 37°C ± 0,5 M 16.05.97 VA 16.05.97

③ ~~100~~ M 16.05.97

③ 75 TPN M 16.05.97 VA 16.05.97

Dissolvant DAP: Solux n°5

FOURNIER 1001712  
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Subject to  
Protective Order

RC 24.06.97

012

3. Pesée des gélules Bal GAL 205

16.05.97 12:16:34  
Code Lot : 48  
Wardle Aero. Canada  
ID

0.0 mg

420.5 mg

ID

0.0 mg

428.6 mg

1 .000 ABS 290.0 NM  
1 .001 ABS 290.0 NM  
1 .000 ABS 290.0 NM

1 T<sub>0</sub> .001 ABS 290.0 NM  
1 .001 ABS 290.0 NM  
1 .000 ABS 290.0 NM  
1 .000 ABS 290.0 NM  
1 .001 ABS 290.0 NM  
1 .001 ABS 290.0 NM

1 T<sub>5</sub> .068 ABS 290.0 NM  
1 .048 ABS 290.0 NM  
1 .096 ABS 290.0 NM  
1 .099 ABS 290.0 NM  
1 .131 ABS 290.0 NM  
1 .052 ABS 290.0 NM

1 T<sub>10</sub> .444 ABS 290.0 NM  
1 .220 ABS 290.0 NM  
1 .496 ABS 290.0 NM  
1 .534 ABS 290.0 NM  
1 .520 ABS 290.0 NM  
1 .402 ABS 290.0 NM

1 T<sub>15</sub> .798 ABS 290.0 NM  
1 .677 ABS 290.0 NM  
1 .832 ABS 290.0 NM  
1 .850 ABS 290.0 NM  
1 .842 ABS 290.0 NM  
1 .754 ABS 290.0 NM

1 T<sub>20</sub> .982 ABS 290.0 NM  
1 .923 ABS 290.0 NM  
1 1.017 ABS 290.0 NM  
1 1.004 ABS 290.0 NM  
1 1.013 ABS 290.0 NM  
1 .951 ABS 290.0 NM

1 T<sub>25</sub> 1.179 ABS 290.0 NM  
1 1.148 ABS 290.0 NM  
1 1.166 ABS 290.0 NM  
1 1.211 ABS 290.0 NM  
1 1.201 ABS 290.0 NM  
1 1.170 ABS 290.0 NM

1 T<sub>30</sub> 1.295 ABS 290.0 NM  
1 1.278 ABS 290.0 NM  
1 1.288 ABS 290.0 NM  
1 1.281 ABS 290.0 NM  
1 1.301 ABS 290.0 NM  
1 1.259 ABS 290.0 NM

1 T<sub>35</sub> 1.343 ABS 290.0 NM  
1 1.331 ABS 290.0 NM  
1 1.329 ABS 290.0 NM  
1 1.352 ABS 290.0 NM  
1 1.356 ABS 290.0 NM  
1 1.321 ABS 290.0 NM

1 T<sub>40</sub> 1.391 ABS 290.0 NM  
1 1.389 ABS 290.0 NM  
1 1.356 ABS 290.0 NM  
1 1.398 ABS 290.0 NM  
1 1.376 ABS 290.0 NM  
1 1.369 ABS 290.0 NM

1 T<sub>45</sub> 1.534 ABS 290.0 NM  
1 1.555 ABS 290.0 NM  
1 1.545 ABS 290.0 NM  
1 1.543 ABS 290.0 NM  
1 1.545 ABS 290.0 NM  
1 1.533 ABS 290.0 NM

6

0.0 mg

418.3 mg

ID

0.0 mg

420.3 mg

M

h. lecture

Ouhon

lironouche GAL 122

FOURNIER 1001713

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PC26 CG 97

012

3. Pesee des gélules Bal GAL 205

16.05.97 Code ID	12:16:34 Lot : 40 Ward, Micro-Canada	1	0.0 mg
ID		2	420.5 mg
ID		3	0.0 mg
ID		4	420.6 mg
ID		5	0.0 mg
ID		6	424.8 mg
ID			0.0 mg
ID			418.3 mg
ID			0.0 mg
ID			420.3 mg

h. Lecture

Union 810, DAP - Chronomètre BAL 122

1	1.391	ABS	290.0	NM
1	1.309	ABS	290.0	NM
1	1.356	ABS	290.0	NM
1	1.398	ABS	290.0	NM
1	1.376	ABS	290.0	NM
1	1.369	ABS	290.0	NM
1	1.534	ABS	290.0	NM
1	1.555	ABS	290.0	NM
1	1.545	ABS	290.0	NM
1	1.503	ABS	290.0	NM
1	1.545	ABS	290.0	NM
1	1.533	ABS	290.0	NM

FOURNIER 1001714

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Protective Order

PC 26.06.97

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
 DATE : 16/05/97  
 APPAREIL : Sotax 5 - Uvikon 810  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2mm

TITRE : gélules lipidil micro 200 Canada lot 40  
 N° CAHIER : LI 178ter dissolution n°2 p 120 M. RL  
 FICHER : m:\commun\glnq\donnbase\li178ter\dissolution\lot 40 Canada  
 ELUANT : LSNa 0,025M  
 AGITATION : 75 TPM

## PRÉPARATION DES ÉCHANTILLONS

masse théorique	200
dosage théorique	200 en mg

masse de la prise d'essai  
 quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
200,00	200,00	200,00	200,00	200,00	200,00
200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

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## SUIVI DE LA DISSOLUTION

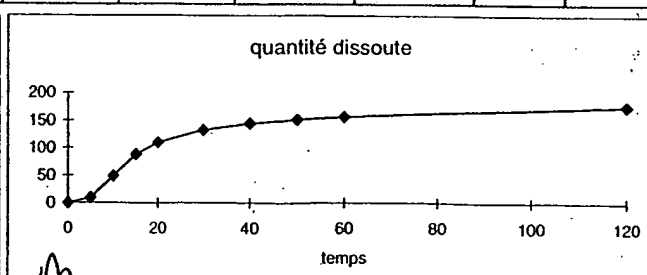
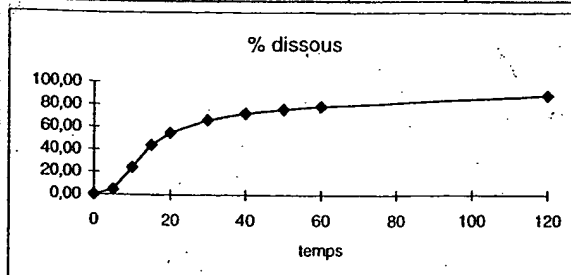
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,068	0,048	0,096	0,099	0,131	0,052
10	1000	0,444	0,22	0,496	0,534	0,52	0,402
15	1000	0,798	0,677	0,832	0,85	0,842	0,754
20	1000	0,982	0,923	1,017	1,004	1,013	0,951
30	1000	1,179	1,148	1,166	1,211	1,201	1,17
40	1000	1,295	1,278	1,288	1,281	1,301	1,259
50	1000	1,343	1,331	1,329	1,352	1,356	1,321
60	1000	1,391	1,389	1,356	1,398	1,376	1,369
120	1000	1,534	1,555	1,545	1,543	1,545	1,533

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	4,57	3,78	2,67	5,33	5,50	7,28	2,89	1,78	38,93
10,0	24,25	24,69	12,24	27,58	29,69	28,93	22,35	6,49	26,78
15,0	44,15	44,48	37,69	46,39	47,40	46,96	42,02	3,74	8,47
20,0	54,90	54,92	51,54	56,90	56,19	56,69	53,17	2,15	3,92
30,0	66,15	66,14	64,30	65,46	67,97	67,42	65,60	1,35	2,04
40,0	72,28	72,91	71,84	72,56	72,19	73,31	70,87	0,86	1,19
50,0	75,69	75,94	75,14	75,19	76,49	76,72	74,66	0,82	1,09
60,0	78,35	78,97	78,73	77,06	79,43	78,21	77,70	0,87	1,11
120,0	87,77	87,31	88,34	87,94	87,87	87,98	87,19	0,44	0,50
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	9,15	7,56	5,33	10,67	11,00	14,56	5,78	3,56	38,93
10,0	48,49	49,37	24,47	55,16	59,39	57,85	44,70	12,98	26,78
15,0	88,31	88,95	75,37	92,77	94,80	93,92	84,03	7,48	8,47
20,0	109,80	109,84	103,08	113,79	112,38	113,39	106,34	4,30	3,92
30,0	132,29	132,27	128,59	130,91	135,94	134,84	131,20	2,70	2,04
40,0	144,56	145,82	143,68	145,12	144,39	146,62	141,74	1,73	1,19
50,0	151,38	151,87	150,27	150,39	152,99	153,45	149,33	1,65	1,09
60,0	156,70	157,95	157,46	154,12	158,85	156,42	155,39	1,74	1,11
120,0	175,54	174,61	176,67	175,88	175,74	175,97	174,38	0,88	0,50
0,0									
0,0									
0,0									



Bo 21.06.97



le 3/6/97.

Dissolution comprimé IF 178 Se Canada.

lot Co 194.

1. Préparation du milieu de dissolution, LSNa 0.25 M.

peser de l'eau sur balance PH 4600 galecs, en petite quantité dans un bœcher de 3 l.

Introduire dans un bœcher de 10 l.

Peser le LSNa correspondant. Mélanger avec eau du bœcher de 10 l.

Peser de l'eau dans un bœcher de 3 l et peser le LSNa correspondant. Mélanger.

Introduire la solution de LSNa des 2 bœchers dans bonbonne de 20 litres. Mélanger.

\* bœcher de 10 litres, eau

03.06.97 08:11:41

642.4 g tare

0.00 g

1908.9 g net

-2551.3 g brut

644.7 g tare

0.00 g

2050.8 g net

-2695.5 g brut

644.4 g tare

0.00 g

2137.1 g net

-2781.6 g brut

644.6 g tare

0.00 g

3059.3 g net

oublié un premier brut AL.

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total 1908,9 + 2050,8 + 2137,1 + 3059,3 = 9156,1 g RE 03.06.97

soit 9,156 kg.

quantité de LsNa Au 1547 à peser pour bête de 101.

015

$$3,156 \times 0,025 \times 288,4 = 66,01 \text{ g.}$$

$$\text{Pt du LsNa} = 288,4 \text{ g.}$$

03.06.97  
Code

08:21:07  
1547

163.59 g raie =

66.01 g net

-229.62 g brut

\*bête de 3 p.

eau.

03.06.97 08:29:12  
644.9 g raie

$$\text{soit } 3,145 \times 288,4 \times 0,025 = 22,68 \text{ g de LsNa à peser.}$$

3144.5 g net

-3789.4 g brut

LsNa.  
03.06.97 08:34:54  
Code 1547

163.74 g raie =

22.69 g net

-186.44 g brut

2. Pesée du milieu de dissolution garobes.

$$1 \text{ p de LsNa } 0,025 \text{H} = 1001,0 \text{ g.}$$

FOURNIER 1001717

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03.06.97 08:42:52  
ID 1

ID

// 3

// ID

5

768.9 g raie

768.2 g raie

760.7 g raie

1001.0 g net

1001.0 g net

1001.0 g net

-1769.9 g brut

oubli' impimer brut

-1761.7 g brut

ID 2

ID

4

ID

6

698.8 g raie

760.9 g raie

753.2 g raie

1001.0 g net

1001.0 g net

1001.0 g net

-1699.8 g brut =

-1762.0 g brut =

-1754.2 g brut =

PC 06.06.97

3. Conditions de dissolution.

dissolveur LORAN AT7 gal 091.

 $T^{\circ} = 37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  AR 3/6/97 SR 03/06/97 $\delta$ : 75 ppm. AR 3/6/97 SR 03/06/974. Pesée des comprimés gal 25.

03.06.97	10:15:55	194	1	0.0000 g
Code			2	0.7139 g
ID			3	0.0000 g
			4	0.7106 g
			5	0.0000 g
			6	0.7113 g
				0.0000 g
				0.7085 g
				0.0000 g
				0.7070 g
				0.0000 g
				0.7139 g

5. Lecturesur spectrophotomètre KONTRON 922 gal 233  
cuves de 2 mm de trajet optique.

LONGUEUR D'ONDE[nm]..... 290.0  
 TEMPS D'ATTENTE [s]..... 0.0  
 TEMPS INTEGRATION [s]..... 5.0  
 NOMBRE D'ECHANTILLONS..... 1  
 MODE CALC..... Non  
 CHANGEMENT LAMPES[nm]..... 340  
 LAMPE DEUTERIUM..... Oui  
 LAMPE TUNGSTENE..... Oui  
 FENTE(S) [nm]..... 2.0  
 IMPRESSION AUTO..... Non  
 SAUVEGARDE AUTO..... Non  
 TRANSFERT AUTO..... Non

KONTRON INSTRUMENTS UVIKON 922

Opérateur

FOURNIER 1001718

 Highly Confidential  
 Subject to  
 Protective Order

AC06.06.97

## DISSOLUTION

m:\commun\gl\q\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
 DATE : 03/06/97  
 APPAREIL : gal 091 233  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2mm

TITRE : Comprimés Canada lot CO194  
 N° CAHIER : LI 178ter dissolution n°2 p14  
 FICHER : m:\commun\gl\q\donnbase\li178ter\dissolution\lot CO194 Canada  
 ELUANT : LNa 0,025M  
 AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001719

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

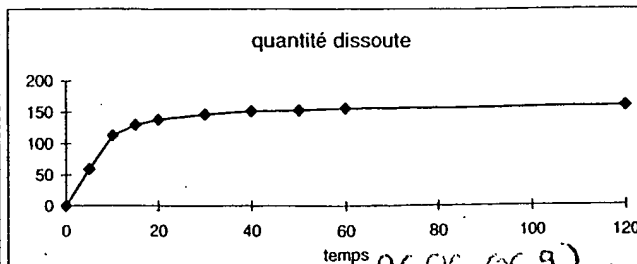
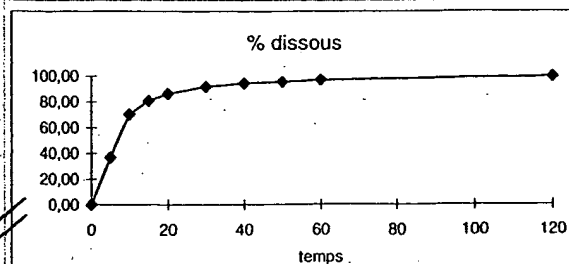
volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
5	1000	0,552	0,424	0,514	0,555	0,58	0,559
10	1000	1,007	0,959	1,012	1,035	1,032	1,017
15	1000	1,173	1,142	1,166	1,161	1,159	1,146
20	1000	1,221	1,229	1,245	1,222	1,223	1,228
30	1000	1,296	1,291	1,301	1,302	1,305	1,302
40	1000	1,339	1,338	1,325	1,336	1,328	1,336
50	1000	1,353	1,34	1,341	1,328	1,342	1,337
60	1000	1,349	1,362	1,368	1,352	1,366	1,35
120	1000	1,374	1,395	1,385	1,408	1,38	1,372

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	36,85	38,33	29,44	35,69	38,54	40,28	38,82	3,92	10,64
10,0	70,35	70,12	66,74	70,46	72,07	71,87	70,82	1,93	2,74
15,0	80,94	82,00	79,79	81,50	81,18	81,05	80,13	0,84	1,03
20,0	86,21	85,74	86,22	87,39	85,82	85,89	86,22	0,61	0,71
30,0	91,61	91,37	90,96	91,71	91,80	92,01	91,79	0,38	0,41
40,0	94,43	94,81	94,67	93,83	94,61	94,06	94,60	0,39	0,41
50,0	95,34	96,25	95,27	95,40	94,52	95,50	95,13	0,56	0,59
60,0	97,04	96,44	97,26	97,74	96,65	97,63	96,50	0,58	0,60
120,0	99,44	98,64	100,03	99,40	101,00	99,07	98,50	0,94	0,95
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	58,96	61,33	47,11	57,11	61,67	64,44	62,11	6,27	10,64
10,0	112,55	112,20	106,79	112,73	115,31	114,99	113,31	3,08	2,74
15,0	129,50	131,20	127,66	130,40	129,88	129,67	128,21	1,34	1,03
20,0	137,94	137,18	137,96	139,83	137,31	137,43	137,96	0,98	0,71
30,0	146,57	146,20	145,53	146,74	146,87	147,22	146,86	0,61	0,41
40,0	151,09	151,69	151,47	150,13	151,38	150,50	151,36	0,62	0,41
50,0	152,55	153,99	152,44	152,65	151,23	152,79	152,22	0,90	0,59
60,0	155,26	154,30	155,62	156,39	154,63	156,21	154,40	0,93	0,60
120,0	159,11	157,83	160,05	159,04	161,61	158,52	157,60	1,51	0,95
0,0									
0,0									
0,0									



RC 06.06.97

## LONGUEUR D ONDE FIXE

			06-03-1997	12.47
Lambda	No.	Valeur_E		
290.0	1	0.0000_1 <i>A2ai/ai</i>	290.0	35 1.2955_1
290.0	2	0.0023_1 <i>1.4516/1.4516</i>	290.0	36 1.2908_1
290.0	3	-0.0001_1 <i>1.42</i>	290.0	37 1.3012_1
290.0	4	-0.0001_1	290.0	38 1.3024_1 <i>30'</i>
290.0	5	-0.0001_1	290.0	39 1.3047_1
290.0	6	-0.0003_1	290.0	40 1.3017_1
290.0	7	-0.0009_1 <i>10</i>	290.0	41 1.3389_1
290.0	8	-0.0010_1	290.0	42 1.3383_1
290.0	9	-0.0003_1	290.0	43 1.3253_1 <i>40'</i>
290.0	10	0.5518_1	290.0	44 1.3363_1
290.0	11	0.4236_1	290.0	45 1.3277_1
290.0	12	0.5135_1 <i>5'</i>	290.0	46 1.3357_1
290.0	13	0.5545_1	290.0	47 1.3530_1
290.0	14	0.5798_1	290.0	48 1.3396_1
290.0	15	0.5592_1	290.0	49 1.3413_1
290.0	16	1.0069_1	290.0	50 1.3276_1 <i>50'</i>
290.0	17	0.9585_1	290.0	51 1.3421_1
290.0	18	1.0120_1	290.0	52 1.3366_1
290.0	19	1.0352_1 <i>10'</i>	290.0	53 1.3490_1
290.0	20	1.0318_1	290.0	54 1.3616_1
290.0	21	1.0165_1	290.0	55 1.3677_1
290.0	22	1.1728_1	290.0	56 1.3523_1 <i>60' =</i>
290.0	23	1.1420_1	290.0	57 1.3659_1
290.0	24	1.1656_1	290.0	58 1.3504_1
290.0	25	1.1614_1 <i>15'</i>	290.0	59 1.3740_1
290.0	26	1.1591_1	290.0	60 1.3947_1
290.0	27	1.1459_1	290.0	61 1.3848_1
290.0	28	1.2212_1	290.0	62 1.4081_1
290.0	29	1.2291_1	290.0	63 1.3800_1 <i>120' =</i>
290.0	30	1.2601_1 <i>gautt</i>	290.0	64 1.3716_1
290.0	31	1.2453_1		
290.0	32	1.2223_1		
290.0	33	1.2227_1 <i>6' =</i>		
290.0	34	1.2276_1		

FOURNIER 1001720

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63/6/97

# Dissolution comprimé CF173 Jca Canada Ar C0197

## 1. Préparation du milieu de dissolution LNE 0,025M

voir p.14.

## 2. Pesée du milieu de dissolution gal 065

1 litre de LNE 0,025M = 1001,0g.

03.06.97	11:54:35				
ID	1	ID	3	ID	5
760.5 g tare		760.9 g tare		768.9 g tare	
1001.0 g net		1001.0 g net		1001.0 g net	
-1761.5 g -tare		-1761.8 g -tare		-1769.9 g -tare	
ID	2	ID	4	ID	6
753.1 g tare		767.1 g tare		698.9 g tare	
1001.0 g net		1001.0 g net		1001.0 g net	
-1754.0 g -tare		-1768.1 g -tare		-1699.9 g -tare	

## 3. Conditions de dissolution

dissolvant SPTX AT7 gal 065

T° = 37°C ± 0,5°C Ar 3/6/97 82 03/06/97

8. 75 rpm Ar 3/6/97 82 03/06/97

## 4. Pesée des comprimés gal 065

FOURNIER 1001721

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RC 06.06.97

03.06.97

13:52:57

ID

1

ID

3

ID

5

0.0000 g

0.0000 g

0.0000 g

0.7106 g

0.7129 g

0.7137 g

ID

2

ID

4

ID

6

0.0000 g

0.0000 g

0.0000 g

0.7157 g

0.7001 g

0.7076 g

S. Leduc

sur spectrophotomètre MONTAN 922 gal 233 avec cuves de 2mm de trajet.

COMPRIMES LF178TER CANADA LOT C0197

06-03-1997 16:14

Lambda No. Valeur\_E

290.0	1	0.0007_1
290.0	2	-0.0001_1
290.0	3	-0.0001_1
290.0	4	-0.0002_1
290.0	5	-0.0002_1
290.0	6	-0.0003_1
290.0	7	-0.0003_1
290.0	8	0.0019_1
290.0	9	0.0000_1
290.0	10	0.3838_1
290.0	11	0.3913_1
290.0	12	0.4987_1
290.0	13	0.2880_1
290.0	14	0.3847_1
290.0	15	0.7308_1
290.0	16	0.9570_1
290.0	17	0.9616_1
290.0	18	1.0086_1
290.0	19	0.8034_1
290.0	20	0.9246_1
290.0	21	1.0422_1
290.0	22	1.1342_1
290.0	23	1.1446_1
290.0	24	1.1469_1
290.0	25	1.0677_1
290.0	26	1.1251_1
290.0	27	1.1819_1
290.0	28	1.2227_1
290.0	29	1.2292_1
290.0	30	1.2335_1
290.0	31	1.1818_1
290.0	32	1.2132_1
290.0	33	1.2397_1
290.0	34	1.2880_1
290.0	35	1.2957_1
290.0	36	1.2916_1
290.0	37	1.2690_1
290.0	38	1.2956_1
290.0	39	1.2966_1

290.0	40	1.3215_1
290.0	41	1.3494_1
290.0	42	1.3204_1
290.0	43	1.3192_1
290.0	44	1.3313_1
290.0	45	1.3366_1
290.0	46	1.3481_1
290.0	47	1.3546_1
290.0	48	1.3378_1
290.0	49	1.3331_1
290.0	50	1.2346_1
290.0	51	1.3494_1
290.0	52	1.3376_1
290.0	53	1.3596_1
290.0	54	1.3409_1
290.0	55	1.2492_1
290.0	56	1.2742_1
290.0	57	1.3520_1
290.0	58	1.3709_1
290.0	59	1.3812_1
290.0	60	1.3484_1
290.0	61	1.3822_1
290.0	62	1.3835_1
290.0	63	1.3897_1

goutte d'essence  
cremeuse Pa  
avec plus de  
produit pour  
refaire une  
lecture.

R. 316/97

FOURNIER 1001722

Highly Confidential  
Subject to  
Protective Order

R. 06 06 97

# DISSOLUTION

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : A GRANDJEAN  
DATE : 03/06/97  
APPAREIL : gal 091 233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : Comprimés Canada lot CO197  
N° CAHIER : Lf 178ter dissolution n°2 p18  
FICHIER : m:\commun\ghq\donnbase\lf178ter\dissolution\lot CO197 Canada  
ELUANT : LSNa 0,025M  
AGITATION : 75 TPM

m:\commun\ghq\traitdon\distem5  
date édition: le 06/02/97

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

masse de la prise d'essai  
quantité de principe actif

FOURNIER 1001723

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Témoins 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

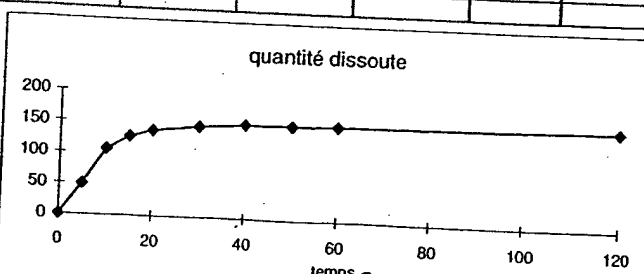
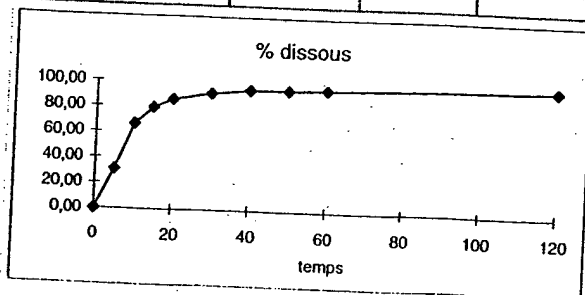
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,384	0,391	0,499	0,288	0,385	0,731
10	1000	0,957	0,962	1,009	0,803	0,925	1,042
15	1000	1,134	1,145	1,147	1,068	1,125	1,182
20	1000	1,223	1,229	1,234	1,182	1,213	1,24
30	1000	1,288	1,296	1,292	1,269	1,296	1,297
40	1000	1,322	1,349	1,32	1,319	1,331	1,337
50	1000	1,348	1,355	1,338	1,333	1,235	1,349
60	1000	1,338	1,36	1,341		1,274	1,352
120	1000	1,371	1,381	1,348	1,382	1,384	1,39

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	31,00	26,67	27,15	34,65	20,00	26,74	50,76	10,74	34,65
10,0	66,10	66,59	66,94	70,24	55,86	64,37	72,61	5,80	8,78
15,0	79,20	79,22	79,98	80,18	74,55	78,58	82,70	2,68	3,38
20,0	85,61	85,79	86,21	86,62	82,83	85,08	87,14	1,53	1,79
30,0	90,86	90,73	91,29	91,07	89,29	91,27	91,53	0,82	0,90
40,0	94,09	93,54	95,42	93,47	93,20	94,15	94,75	0,86	0,92
50,0	94,32	95,80	96,31	95,17	94,63	97,94	96,05	3,18	3,37
60,0	95,27	95,58	97,13	95,85		91,08	96,73	2,43	2,55
120,0	98,61	98,33	99,06	96,80	98,49	99,16	99,84	1,04	1,05
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	49,59	42,67	43,44	55,44	32,00	42,78	81,22	17,18	34,65
10,0	105,77	106,55	107,11	112,39	89,38	102,99	116,18	9,28	8,78
15,0	126,72	126,75	127,97	128,28	119,27	125,73	132,32	4,28	3,38
20,0	136,98	137,26	137,94	138,59	132,53	136,13	139,42	2,45	1,79
30,0	145,38	145,17	146,07	145,72	142,86	146,03	146,44	1,31	0,90
40,0	150,54	149,66	152,68	149,55	149,12	150,64	151,61	1,38	0,92
50,0	150,91	153,28	154,10	152,28	151,41	140,71	153,68	5,09	3,37
60,0	152,43	152,92	155,40	153,36		145,73	154,77	3,88	2,55
120,0	157,78	157,33	158,49	154,88	157,59	158,66	159,74	1,66	1,05
0,0									
0,0									
0,0									



RC 06 06 97



25/6/87

021

Dissolution Compurac UF 178 Jec Canada

lot CO 200,

1. Préparation du milieu de dissolution LSWA 0,025%

balance gabo5.

pesée de l'eau :

05.06.97 08:50:05

643.1 g tare

2609.2 g net

-3252.3 g -net

soit un total de  $2609,2 + 2271,3 + 1655,6 =$

6,536 kg.

soit  $6,536 \times 288,4 \times 0,025 = 47,1$  g de LSW

à peser.

645.6 g tare

2271.3 g net

-2916.9 g -net

PH = 288,4

647.9 g tare

1655.6 g net

-2303.3 g -net.

pesée du LSW.

05.06.97 10:45:04

Code AM 1547

0.00 g

90.53 g tare

0.00 g

47.10 g net

0.00 g

RC 137.63 g brut

FOURNIER 1001724

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Protective Order

REC06.97

U.S.A.

## 2 - Pesée du milieu de dissolution - GAL065

1 l de L8Na 0.025H pèse 1001,0g -

05.06.97

11:09:15

Code

1

Code

3

Code

5

0.00 g

0.00 g

0.00 g

768.8 g Tare

768.5 g Tare

767.5 g Tare

0.00 g

0.00 g

0.00 g

1001.0 g Net

1001.0 g Net

1001.0 g Net

0.00 g

0.00 g

0.00 g

1769.8 g Bulk

1761.4 g Bulk

1768.5 g Bulk

Code

2

Code

4

Code

6

0.00 g

0.00 g

0.00 g

768.8 g Tare

753.1 g Tare

698.8 g Tare

0.00 g

0.00 g

0.00 g

1001.0 g Net

1001.0 g Net

1001.0 g Net

0.00 g

0.00 g

~~0.01 g~~

1761.8 g Bulk

1754.1 g Bulk

0.00 g

RC

RC

1699.8 g Bulk

RC

## 3 - Conditions de dissolution

dissolutes SOTAX AT7 - GAL091

Temperature 37°C ± 0,5°C RC 05.06.97 AG S/G/97

Vitesse 75TPM

RC 05.06.97 AG S/G/97

## 4. Pesée des comprimés - GAL205

FOURNIER 1001725

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Subject to  
Protective Order

05.06.97 14:06:50

Code 178-0200

Code 1

0.0000 g

0.7148 g

Code 2

0.0000 g

0.7149 g

Code 3

0.0000 g

0.7089 g

Code 4

0.0000 g

0.7088 g

Code 5

0.0000 g

0.7089 g

Code 6

0.0000 g

0.6993 g

AG 6/6/97 RC

5. lecture

Sur Spectrophotomètre Kontron 922 GFL 233 en cuves de 2mm de trajet

## LONGUEUR D ONDE FIXE

Liste Paramètre

06-05-1997 14:18

LONGUEUR D'ONDE[nm]..... 290.0  
 TEMPS D'ATTENTE [s]..... 0.0  
 TEMPS INTEGRATION [s]..... 5.0  
 NOMBRE D'ECHANTILLONS..... 1

MODE CALC..... Non

CHANGEMENT LAMPES[nm]..... 340  
 LAMPE DEUTERIUM..... Oui  
 LAMPE TUNGSTENE..... Oui  
 FENTE(S) [nm]..... 2.0  
 IMPRESSION AUTO..... Non  
 SAUVEGARDE AUTO..... Non  
 TRANSFERT AUTO..... Non

KONTRON INSTRUMENTS UVIKON 922

Opérateur

RC

## LONGUEUR D ONDE FIXE 290NM 75TPM LF178TER LOT C0200

06-05-1997 17:11

Lambda	No.	Valeur_E
290.0	1	0.0000_1 <i>Ain/Ain</i>
290.0	2	0.0010_1 <i>LSNa/LSNa</i>
290.0	3	0.0013_1
290.0	4	0.0014_1
290.0	5	0.0011_1 <i>0 mm</i>
290.0	6	0.0009_1
290.0	7	0.0027_1
290.0	8	0.0009_1
290.0	9	0.4201_1
290.0	10	0.4382_1
290.0	11	0.4639_1 <i>5 mm</i>
290.0	12	0.5384_1
290.0	13	0.5663_1
290.0	14	0.4676_1
290.0	15	0.9829_1
290.0	16	0.9865_1
290.0	17	0.9874_1 <i>10 mm</i>
290.0	18	1.0207_1
290.0	19	1.0272_1
290.0	20	0.9509_1
290.0	21	1.1464_1
290.0	22	1.1473_1
290.0	23	1.1415_1 <i>15 mm</i>
290.0	24	1.1502_1
290.0	25	1.1504_1
290.0	26	1.1044_1
290.0	27	1.2258_1
290.0	28	1.2266_1
290.0	29	1.2108_1 <i>20 mm</i>
290.0	30	1.2243_1
290.0	31	1.2177_1
290.0	32	1.1815_1
290.0	33	1.2948_1
290.0	34	1.2984_1
290.0	35	1.2802_1 <i>30 mm</i>
290.0	36	1.2877_1
290.0	37	1.2801_1
290.0	38	1.2574_1
290.0	39	1.3204_1
290.0	40	1.3052_1
290.0	41	1.3247_1 <i>40 mm</i>
290.0	42	1.3117_1
290.0	43	1.3081_1
290.0	44	1.2897_1
290.0	45	1.3275_1
290.0	46	1.3421_1
290.0	47	1.3348_1 <i>50 mm</i>
290.0	48	1.3313_1
290.0	49	1.3238_1
290.0	50	1.3154_1
290.0	51	1.3604_1
290.0	52	1.3503_1
290.0	53	1.3457_1 <i>60 mm</i>
290.0	54	1.3388_1
290.0	55	1.3345_1
290.0	56	1.3189_1
290.0	57	1.3738_1
290.0	58	1.3758_1
290.0	59	1.3693_1 <i>120 mm</i>
290.0	60	1.3788_1
290.0	61	1.3761_1
290.0	62	1.3559_1

FOURNIER 1001726

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 Protective Order

RC-6/6/97

# DISSOLUTION

m:\commun\lng\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 05/06/97  
APPAREIL : GAL091 GAL233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2 mm

TITRE : Comprimés Canada lot C0200  
N° CAHIER : LF178TER dissolution N°2 p 021  
FICHIER : m:\commun\lng\donnbase\dissolution\lotC0200 Canada  
ELUANT : LNa 0,025M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Fournier 1001727

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

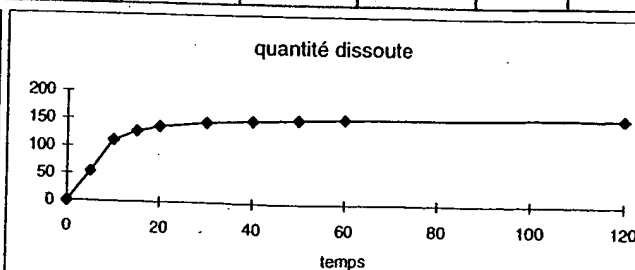
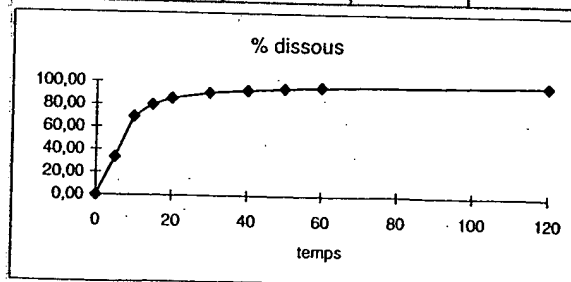
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,42	0,438	0,464	0,538	0,566	0,468
10	1000	0,983	0,987	0,987	1,021	1,027	0,951
15	1000	1,146	1,147	1,142	1,15	1,15	1,104
20	1000	1,226	1,227	1,211	1,224	1,218	1,182
30	1000	1,295	1,298	1,28	1,288	1,28	1,257
40	1000	1,32	1,305	1,325	1,312	1,308	1,29
50	1000	1,328	1,342	1,335	1,331	1,324	1,315
60	1000	1,36	1,35	1,346	1,339	1,335	1,319
120	1000	1,374	1,376	1,369	1,379	1,376	1,356

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	33,50	29,17	30,42	32,22	37,36	39,31	32,50	3,99	11,91
10,0	69,10	68,41	68,69	68,70	71,09	71,52	66,20	1,95	2,82
15,0	79,67	80,07	80,15	79,81	80,40	80,41	77,16	1,25	1,57
20,0	85,26	86,02	86,10	85,00	85,94	85,54	82,96	1,20	1,41
30,0	90,43	91,24	91,46	90,21	90,81	90,26	88,58	1,04	1,15
40,0	92,75	93,43	92,39	93,78	92,92	92,65	91,31	0,87	0,94
50,0	94,53	94,44	95,42	94,93	94,70	94,22	93,49	0,66	0,70
60,0	95,85	97,12	96,44	96,16	95,72	95,44	94,22	0,99	1,03
120,0	98,41	98,57	98,71	98,23	98,96	98,75	97,25	0,62	0,63
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	53,59	46,67	48,67	51,56	59,78	62,89	52,00	6,38	11,91
10,0	110,56	109,46	109,91	109,92	113,74	114,43	105,93	3,12	2,82
15,0	127,47	128,11	128,24	127,70	128,64	128,66	123,46	2,00	1,57
20,0	136,42	137,64	137,76	136,00	137,51	136,86	132,74	1,92	1,41
30,0	144,68	145,99	146,33	144,34	145,30	144,42	141,73	1,66	1,15
40,0	148,40	149,48	147,83	150,05	148,68	148,25	146,09	1,39	0,94
50,0	151,25	151,11	152,67	151,89	151,52	150,75	149,58	1,05	0,70
60,0	153,36	155,40	154,30	153,86	153,15	152,71	150,76	1,58	1,03
120,0	157,46	157,71	157,94	157,16	158,34	158,00	155,60	0,99	0,63
0,0									
0,0									
0,0									



AB-6/6/97

08.06.97

025

Dissolution Comprimés LF 178 Ter

lot PK #158 à 100mg de Feno

Milieu LSNa 0,025N à 75 TPN

1 Préparation milieu de dissolution Balance GAL 065

Pesée de l'eau:

EAU

09.06.97 09:50:42

0.00 g

229.59 g Tare

0.00 g

3008.7 g Net

0.00 g

-3238.3 g - Brut

0.00 g

229.61 g Tare

0.00 g

2942.5 g Net

0.00 g

-3172.1 g - Brut

0.00 g

229.59 g Tare

0.00 g

2473.2 g Net

-0.01 g

-2702.8 g - Brut

Total de l'eau pesée:

$$3008,7 + 2942,5 + 2473,2 = 8424,4 \text{ g.}$$

Pour un milieu à 0,025N:

masse de LSNa à peser (PN 288,4 g)

$$8,4244 \times 288,4 \times 0,025 = 60,7 \text{ g.}$$

LSNa ARR 1547

09.06.97 10:08:11

0.00 g

143.23 g Tare

0.00 g

60.71 g Net

0.00 g

-203.94 g - Brut

FOURNIER 1001728

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08.06.97

026

2. Pesée du milieu de dissolution Bal. GAL0651 litre de l'Na 0,025 N  $\rightarrow$  1001,0 g.

09.06.97	11:05:31				
1	0.00 g	3	0.00 g	5	-0.01 g
699.3 g	Tare	760.5 g	Tare	753.0 g	Tare
0.00 g		0.00 g		0.00 g	
1001.0 g	Net	1001.0 g	Net	1001.0 g	Net
0.00 g		0.00 g		0.00 g	
-1700.4 g	-Brut	-1761.5 g	-Brut	-1754.0 g	-Brut
2	0.00 g	4	0.00 g	6	-0.01 g
767.8 g	Tare	768.8 g	Tare	760.8 g	Tare
0.00 g		0.00 g		0.00 g	
1001.0 g	Net	1001.0 g	Net	1001.0 g	Net
0.00 g		0.01 g		0.00 g	
-1768.8 g	-Brut	-1769.8 g	-Brut	-1761.8 g	-Brut

3. Conditions de dissolution

Solax GAL091

T = 37°C  $\pm$  0,5 M 09.06.97  $\approx$  08/06/97Q = 75 TPN M 09.06.97  $\approx$  08/06/97h. Pesée des comprimés Bal. GAL20509.06.97 12:31:27  
Code lot 158

0.0 mg
554.6 mg
0.0 mg
555.8 mg
0.0 mg
559.0 mg
0.0 mg
561.3 mg
0.0 mg
564.0 mg
0.0 mg
564.9 mg

FOURNIER 1001729

Highly Confidential  
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AC 24.06.97

S. Lecture

Spectrophotomètre GAL 233 - Chronomètre GAL M4

Cuvette 2mm à 290nm

LF 178 TER LOT 158 LSNA 0.025M 75 TPM

06-09-1997 15:14

Lambda No. Valeur\_E

290.0	1	0.0021_1
290.0	2	0.0015_1
290.0	3	0.0014_1
290.0	4	0.0021_1
290.0	5	0.0025_1
290.0	6	0.0029_1
290.0	7	0.4949_1
290.0	8	0.5380_1
290.0	9	0.5231_1
290.0	10	0.4994_1
290.0	11	0.5009_1
290.0	12	0.5074_1
290.0	13	0.6881_1
290.0	14	0.7172_1
290.0	15	0.7077_1
290.0	16	0.7061_1
290.0	17	0.7063_1
290.0	18	0.7112_1
290.0	19	0.7362_1
290.0	20	0.7538_1
290.0	21	0.7544_1
290.0	22	0.7518_1
290.0	23	0.7472_1
290.0	24	0.7589_1
290.0	25	0.7729_1
290.0	26	0.7826_1
290.0	27	0.7901_1
290.0	28	0.7943_1
290.0	29	0.7888_1
290.0	30	0.7838_1
290.0	31	0.8060_1
290.0	32	0.8110_1
290.0	33	0.8131_1
290.0	34	0.8156_1
290.0	35	0.8161_1
290.0	36	0.8154_1
290.0	37	0.8283_1
290.0	38	0.8316_1
290.0	39	0.8461_1
290.0	40	0.8301_1
290.0	41	0.8421_1
290.0	42	0.8421_1
290.0	43	0.8286_1
290.0	44	0.8358_1
290.0	45	0.8493_1
290.0	46	0.8524_1
290.0	47	0.8551_1
290.0	48	0.8456_1
290.0	49	0.8291_1
290.0	50	0.8295_1
290.0	51	0.8460_1
290.0	52	0.8554_1
290.0	53	0.8507_1
290.0	54	0.8552_1

Lambda No. Valeur\_E

290.0 1 0.0001\_1

290.0 2 0.0007\_1

M

Autogaro Air/Air

Autogaro LSNA

FOURNIER 1001730

Highly Confidential  
Subject to  
Protective Order

RC 24.06.97

028

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
 DATE : 06/09/97  
 APPAREIL : gal091gal233  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2mm

TITRE : LF 178ter comprimés PK lot 158  
 N° CAHIER : LI 178ter dissolution n°2 p.25-RL  
 FICHER : m:\commun\glnq\donnbase\LI178ter\dissolution\lot PK 158  
 ELUANT : LSNa 0,025M  
 AGITATION : 75 TPM

0,025N 75TPM  
 12.03.06.97

## PREPARATION DES ECHANTILLONS

masse théorique	100
dosage théorique	100 en mg

masse de la prise d'essai  
 quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
100,00	100,00	100,00	100,00	100,00	100,00
100,00	100,00	100,00	100,00	100,00	100,00

FOURNIER 1001731

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Témoin 100mg/ 0,900

## SUIVI DE LA DISSOLUTION

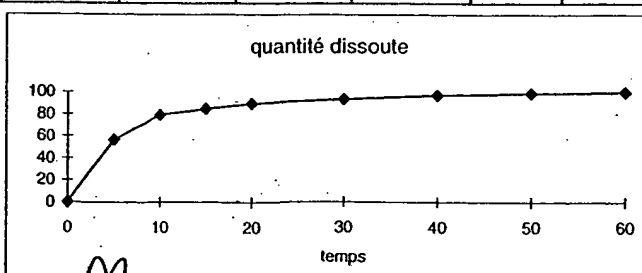
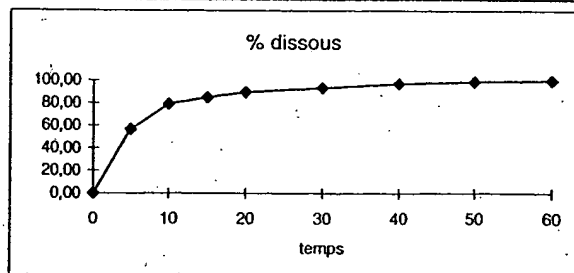
volume prélevé en ml		10					
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
5	1000	0,495	0,538	0,523	0,499	0,501	0,507
10	1000	0,688	0,717	0,708	0,706	0,706	0,711
15	1000	0,736	0,754	0,754	0,752	0,747	0,759
20	1000	0,773	0,783	0,79	0,794	0,789	0,784
30	1000	0,806	0,811	0,813	0,816	0,816	0,815
40	1000	0,828	0,832	0,846	0,83	0,842	0,842
50	1000	0,829	0,836	0,849	0,852	0,855	0,846
60	1000	0,829	0,83	0,846	0,855	0,851	0,855

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
5,0	56,72	55,00	59,78	58,11	55,44	55,67	56,33	1,85	3,26
10,0	79,01	76,99	80,26	79,25	79,00	79,00	79,56	1,10	1,39
15,0	84,72	83,09	85,17	85,15	84,89	84,34	85,69	0,91	1,07
20,0	89,46	88,02	89,23	89,98	90,40	89,84	89,31	0,83	0,93
30,0	93,37	92,55	93,21	93,42	93,72	93,71	93,62	0,45	0,48
40,0	96,92	95,89	96,45	97,99	96,19	97,51	97,53	0,86	0,89
50,0	98,72	96,92	97,82	99,26	99,55	99,89	98,91	1,14	1,15
60,0	99,64	97,84	98,08	99,87	100,83	100,40	100,85	1,36	1,36
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
5,0	56,72	55,00	59,78	58,11	55,44	55,67	56,33	1,85	3,26
10,0	79,01	76,99	80,26	79,25	79,00	79,00	79,56	1,10	1,39
15,0	84,72	83,09	85,17	85,15	84,89	84,34	85,69	0,91	1,07
20,0	89,46	88,02	89,23	89,98	90,40	89,84	89,31	0,83	0,93
30,0	93,37	92,55	93,21	93,42	93,72	93,71	93,62	0,45	0,48
40,0	96,92	95,89	96,45	97,99	96,19	97,51	97,53	0,86	0,89
50,0	98,72	96,92	97,82	99,26	99,55	99,89	98,91	1,14	1,15
60,0	99,64	97,84	98,08	99,87	100,83	100,40	100,85	1,36	1,36
0,0									
0,0									
0,0									
0,0									



RC 24.06.97



10.08.96  
86

029

Dissolution comprimés IF 178 Ter  
lot PK 158 à 100 mg de Feno

Milieu L3Na 0,05 N - 120 TPN

1. Préparation milieu de dissolution Bal GAL 065  
Pesée de l'eau

EAU

10.06.97 09:47:10

0.00 g

229.62 g Tare

0.00 g

2873.6 g Net

0.00 g

-3183.2 g Brut

0.00 g

229.57 g Tare

0.00 g

2661.9 g Net

0.01 g

-2891.5 g Brut

0.00 g

229.58 g Tare

0.00 g

2628.4 g Net

0.00 g

-2857.9 g Brut

M

o Total de l'eau pesée :

$$2873,6 + 2661,9 + 2628,4 = 8163,9 \text{ g}$$

o Pesée du L3Na :

pour un milieu à 0,05 N

CPI: 288,4)

$$\Rightarrow 8,1639 \times 288,4 \times 0,05 = 117,7 \text{ g}$$

L3Na

10.06.97  
Code

ARR

10:01:50  
1547

0.00 g

143.22 g Tare

0.00 g

117.70 g Net

0.00 g

-260.93 g Brut

M

FOURNIER 1001732

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Protective Order

RC 24.06.97



5 lecture

spectrophotomètre GAL 233 - Chromomètre GAL 124

Cave 2mm - 290 nm

LF 178 TER LOT 158 LSNA 0.05 M 120 TPM

06-10-1997 14:17

Lambda No. Valeur\_E

290.0	1	-0.0005_1
290.0	2	0.0000_1
290.0	3	0.0015_1
290.0	4	0.0004_1
290.0	5	0.0009_1
290.0	6	0.0005_1
290.0	7	0.5942_1
290.0	8	0.6671_1
290.0	9	0.6658_1
290.0	10	0.6676_1
290.0	11	0.6985_1
290.0	12	0.7258_1
290.0	13	0.8430_1
290.0	14	0.8153_1
290.0	15	0.8403_1
290.0	16	0.8331_1
290.0	17	0.8307_1
290.0	18	0.8307_1
290.0	19	0.8866_1
290.0	20	0.8517_1
290.0	21	0.8838_1
290.0	22	0.8758_1
290.0	23	0.8721_1
290.0	24	0.8610_1
290.0	25	0.9038_1
290.0	26	0.8675_1
290.0	27	0.8993_1
290.0	28	0.8886_1
290.0	29	0.8794_1
290.0	30	0.8751_1

Lambda No. Valeur\_E

290.0	1	-0.0000_1
290.0	2	-0.0008_1

① Autogaz Air/Air

② LSNa / LSNa

FOURNIER 1001734

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Protective Order

RG 24.06.97

## DISSOLUTION

m:\commun\glnq\traidon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
DATE : 09/09/97  
APPAREIL : gal091gal233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : LF 178ter comprimés PK lot 158 - 0,05M 120 tpm  
N° CAHIER : LI 178ter dissolution n°2 p. 2059  
FICHER : m:\commu\gln\donnbase\LI178ter\dissolution\lot PK 158 0,05M 120 tpm  
ELUANT : 0,05M  
AGITATION : 120 tpm

## PREPARATION DES ECHANTILLONS

masse théorique	100
dosage théorique	100 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	100,00	100,00	100,00	100,00	100,00	100,00
quantité de principe actif	100,00	100,00	100,00	100,00	100,00	100,00

**FOURNIER 1001735**

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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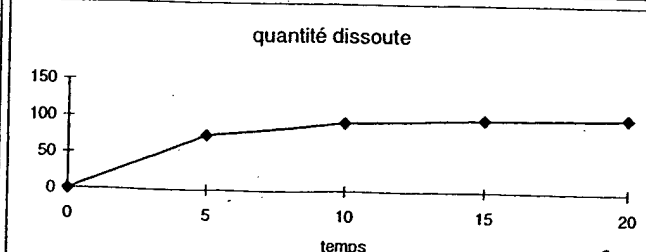
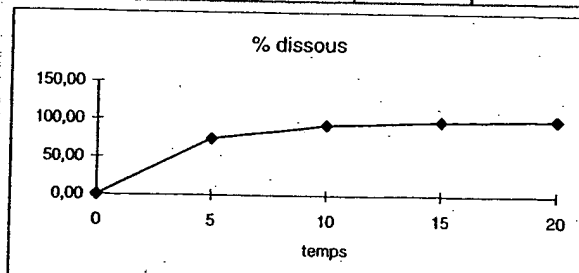
### SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOLTE

[illegible]

RC 09,07,97

11.06.97

Dissolution de comprimés LF 178 rev

033

~~Lot PK 158 à 100 mg de feno M. 11.06.97~~

Lot C0194

Milieu L3Na 0.1N - ~~12077A M. 11.06.97~~

907PN

~~2 comprimés par bol de dissolution - M. 11.06.97~~1. Préparation du milieu de dissolution Bol. GAL 065

Pesée de l'eau

EAU

11.06.97 09:09:42

0.00 g

229.59 g Tare

0.00 g

2919.5 g Net

0.00 g

-3149.1 g -Brut

0.00 g

229.56 g Tare

0.00 g

2780.3 g Net

0.00 g

-3009.9 g -Brut

0.00 g

229.57 g Tare

0.00 g

2673.1 g Net

0.01 g

-2902.6 g -Brut

o Total de l'eau pesée:

$$2919.5 + 2780.3 + 2673.1 = 8372.9 \text{ g}$$

o Pesée du L3Na

pour un milieu à 0.1N

$$C_{PN} = 288.4 \text{ g}$$

$$\rightarrow 8,3729 \times 288.4 \times 0.1 = 241.5 \text{ g}$$

L3Na

11.06.97 09:36:31

Code ARR 1547

0.00 g

143.23 g Tare

0.00 g

241.50 g Net

0.00 g

-384.71 g -Brut

FOURNIER 1001736

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PC-24.06.97

## g. Pesée du milieu de dissolution Bal. GAL 065

1 Lité LBA 0.1N → 1004.0 g.

11.06.97 10:28:15

1 0.00 g

760.6 g Tar

0.00 g

1004.0 g Net

0.00 g

-1764.6 g -Brut

2 0.00 g

767.2 g Tar

0.00 g

1004.0 g Net

0.01 g

-1771.2 g -Brut M

3 0.00 g

698.8 g Tar

0.00 g

1004.0 g Net

0.00 g

-1702.8 g -Brut

4 0.00 g

768.8 g Tar

0.00 g

1004.0 g Net

0.00 g

-1772.8 g -Brut M

5 0.00 g

760.8 g Tar

0.00 g

1004.0 g Net

0.00 g

-1764.8 g -Brut

6 0.00 g

753.0 g Tar

0.00 g

1004.0 g Net

0.00 g

-1757.1 g -Brut M

## 3. Conditions de dissolution

Dissolubest GAL 091

T° 37°C ± 0.5 M 11.06.97 Sz 11/06/92

○ 120 TPN  
M 11.06.97

90 TPN M 11.06.97  
Sz 11/06/92

## h. Pesée des comprimés Bal. GAL 205

11.06.97 12:24:17  
Code  
C0194

1

0.0 mg

707.1 mg

2

0.0 mg

705.9 mg

3

0.0 mg

714.6 mg

4

0.0 mg

703.9 mg

5

0.0 mg

705.9 mg

6

0.0 mg

699.8 mg

S. Lechue

035

Spectrophotomètre GAZ 233 Chronomètre GAI 124

Cuve 2mm - 280nm

M 11.06.97

LF 178 TER LOT 188 LSNA 0.1 M 90 TPM

C 0134

06-11-1997 13:32

Lambda No. Valeur\_E

290.0	1	0.0011_1
290.0	2	0.0010_1
290.0	3	0.0020_1
290.0	4	0.0016_1
290.0	5	0.0004_1
290.0	6	0.0004_1
290.0	7	1.3391_1
290.0	8	1.3739_1
290.0	9	1.3445_1
290.0	10	1.3405_1
290.0	11	1.3505_1
290.0	12	1.2375_1
290.0	13	1.4323_1
290.0	14	1.4347_1
290.0	15	1.4299_1
290.0	16	1.4147_1
290.0	17	1.4206_1
290.0	18	1.3987_1
290.0	19	1.4410_1
290.0	20	1.4423_1
290.0	21	1.4444_1
290.0	22	1.4197_1
290.0	23	1.4273_1
290.0	24	1.4109_1
290.0	25	1.4374_1
290.0	26	1.4430_1
290.0	27	1.4439_1
290.0	28	1.4191_1
290.0	29	1.4274_1
290.0	30	1.4124_1

① 290.0 1 0.0000\_1

② 290.0 2 0.0004\_1

① Autogew Air / Air

② LSNA / LSNA

FOURNIER 1001738

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RC 24.06 97

## DISSOLUTION

m:\commun\ling\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D. LECRIT  
DATE : 11/09/97  
APPAREIL : gal091gal233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2mm

TITRE : LF 178ter comprimés lot C0194-0,1M 90 tpm  
N° CAHIER : LI 178ter dissolution n°2 p 3  
FICHER : m:\commun\lmg\donnbase\lf178ter\dissolution\lot C0194 0,1M 90tpm  
ELUANT : LSNa 0,1M  
AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001739

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Témoin 100mg/l	0,900
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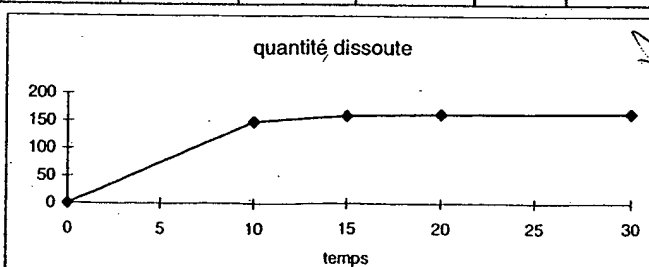
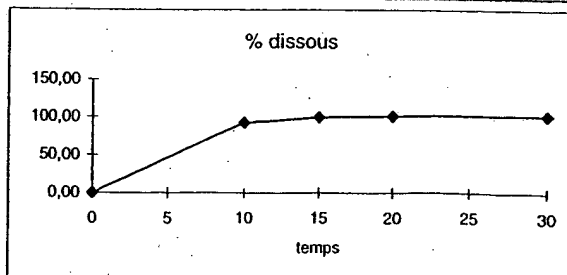
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOUTE

[illegible]

BC 24 06 97



12.06.97

037

Dissolution de comprimés LF178TERLot C0197 - 160 mgLSNa 0,1 M - 90 TPM1 - Préparation du milieu de dissolution. Balance GAL065.

12.06.97 10:20:14

0.00 g Eau purifiée

642.4 g Tare

0.00 g

2700.3 g Net

0.00 g

3342.7 g Brut

0.00 g

644.2 g Tare

0.00 g

2795.1 g Net

0.00 g

3439.2 g Brut

0.00 g

644.2 g Tare

0.00 g

2444.0 g Net

0.00 g

3088.3 g Brut

RC

Total de l'eau pesée

$$2700,3 + 2795,1 + 2444,0 = 7939,4 \text{ g}$$

 Pesée du LSNa pour un milieu  
à 0,1 M PH LSNa = 288,4 g

$$\frac{7939,4 \times 288,4 \times 0,1}{1000} = 228,97 \text{ g}$$

12.06.97 LSNa 10:34:46  
Code ARR 1547

0.00 g

226.36 g Tare

0.00 g

228.97 g Net /

0.00 g

455.33 g Brut

RC

2 - Pesée de bob de dissolution. Balance GAL065.

un litre de LSNa 0,1 M pèse 1004,0 g.

FOURNIER 1001740

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

U 300

Code

11 16

1

Code

3

Code

5

0.00 g

760.5 g Tare

0.00 g

1004.0 g Net

0.00 g

1764.4 g Brut

Code

2

0.00 g

760.8 g Tare

0.00 g

1004.0 g Net

0.00 g

1764.8 g Brut  
RC

0.00 g

698.8 g Tare

0.00 g

1004.0 g Net

0.00 g

1702.8 g Brut

Code

4

0.00 g

767.0 g Tare

0.00 g

1004.0 g Net

0.00 g

1771.0 g Brut  
RC

0.00 g

753.0 g Tare

0.00 g

1004.0 g Net

0.00 g

1757.1 g Brut

Code

6

0.00 g

768.8 g Tare

0.00 g

1004.0 g Net

0.00 g

1772.8 g Brut  
RC3 - Conditions de dissolution

Dissolutes GAL 091 - Soho AT7

Température  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  RC 12.06.97 AE 12/6/97

Vitesse : 90 TPM RC 12.06.97 AE 12/6/97

4 - Pesée des comprimés - Balance GAL 20512.06.97 LER 13:50:50  
Code

Lot C 0197

0.0 mg

712.7 mg

0.0 mg

714.4 mg

0.0 mg

716.2 mg

0.0 mg

714.3 mg

0.0 mg

720.4 mg

0.0 mg

717.8 mg

RC

FOURNIER 1001741

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Subject to  
Protective Order

ML 10.07.97

5. lectures

Sur spectrophotomètre UVKON 922 GAL 233 en cuve de 2mm  
de trajet optique à 290 nm

LONGUEUR D ONDE FIXE 290 NM 2MM LF178TER LOT C0197 LSNA 0.1M 90 TPM /

06-12-1997 14:47

Lambda	No.	Valeur_E
290.0	1	0.0001_1 <i>Au / Au</i>
290.0	2	-0.0048_1 <i>LSNa / LSNa</i>
290.0	3	-0.0036_1
290.0	4	-0.0036_1
290.0	5	-0.0036_1 <i>0mm</i>
290.0	6	-0.0038_1
290.0	7	-0.0034_1
290.0	8	-0.0039_1
290.0	9	1.3206_1
290.0	10	1.2586_1
290.0	11	1.2128_1 <i>10 mm</i>
290.0	12	1.3092_1
290.0	13	1.2957_1
290.0	14	1.3552_1
290.0	15	1.4150_1
290.0	16	1.4074_1
290.0	17	1.4308_1 <i>15 mm</i>
290.0	18	1.4126_1
290.0	19	1.4171_1
290.0	20	1.4395_1
290.0	21	1.4249_1
290.0	22	1.4265_1
290.0	23	1.4557_1 <i>20 mm</i>
290.0	24	1.4267_1
290.0	25	1.4310_1
290.0	26	1.4444_1
290.0	27	1.4270_1
290.0	28	1.4242_1
290.0	29	1.4575_1 <i>30 mm</i>
290.0	30	1.4213_1
290.0	31	1.4301_1
290.0	32	1.4399_1

KONTRON INSTRUMENTS UVIKON 922

Opérateur *RC* .....

FOURNIER 1001742

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Subject to  
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*10.07.97*

## DISSOLUTION

m:\commun\lng\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
 DATE : 12/06/97  
 APPAREIL : GAL091GAL233  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2 mm

TITRE : LF178TER comprimés lot C0197  
 N° CAHIER : LF178TER dissolution n°2 page 037  
 FICHIER : m:\commun\lng\donbase\lf178ter\dissolution\lotC0197 0,1M 90TPM  
 ELUANT : LNa 0,1M  
 AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160 /  
 dosage théorique 160 en mg

FOURNIER 1001743

Highly Confidential

Subject to

Protective Order

masse de la prise d'essai  
 quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
10	1000	1,321 /	1,259 /	1,213 /	1,309 /	1,296 /	1,355 /
15	1000	1,415 /	1,407 /	1,431 /	1,413 /	1,417 /	1,44 /
20	1000	1,425 /	1,427 /	1,456 /	1,427 /	1,431 /	1,444 /
30	1000	1,427 /	1,424 /	1,458 /	1,421 /	1,43 /	1,44 /

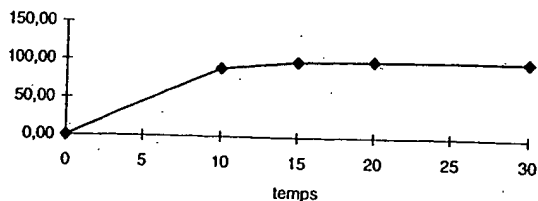
## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	89,73	91,74	87,43	84,24	90,90	90,00	94,10	3,46	3,86
15,0	99,09	98,72	98,15	99,80	98,58	98,85	100,47	0,87	0,87
20,0	100,59	99,91	100,02	102,03	100,04	100,32	101,25	0,86	0,85
30,0	100,98	100,54	100,31	102,67	100,12	100,74	101,47	0,95	0,94
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									

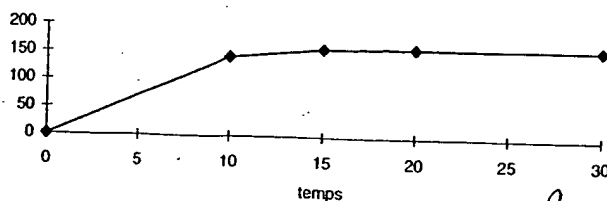
## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	143,57	146,78	139,89	134,78	145,44	144,00	150,56	5,54	3,86
15,0	158,55	157,96	157,03	159,67	157,73	158,16	160,75	1,39	0,87
20,0	160,95	159,85	160,04	163,25	160,07	160,51	162,00	1,37	0,85
30,0	161,56	160,87	160,50	164,28	160,19	161,19	162,36	1,53	0,94
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									

% dissous



quantité dissoute



M-10.07.97

RC

12.06.97

041

Dissolution de comprimés LF 178TERlot C0200 - 160mgLSNa 0,1 M - 90 TPM1 - Préparation du milieu de dissolution - Balance GALOGS

12.06.97 17:09:22

0.00 g Eau purifiée

642.4 g Tare

0.00 g

2644.7 g Net

0.00 g

3287.1 g Brut

0.00 g

644.3 g Tare

0.00 g

2810.8 g Net

0.00 g

3455.1 g Brut

0.00 g

644.2 g Tare

~~0.01 g~~

0.00 g

2439.0 g Net

0.00 g

3083.1 g Brut

RC

Total de l'eau pesée

$$2644,7 + 2810,8 + 2439,0 = 7894,5 \text{ g}$$

Pesée du LSNa pour un milieu

à 0,1 M -  $PM_{LSNa} = 288,4 \text{ g}$ 

$$\frac{7894,5}{1000} \times 288,4 \times 0,1 = 227,68 \text{ g}$$

12.06.97 LSNa 17:22:52  
Code AHR 1547

0.00 g

223.96 g Tare

0.00 g

227.68 g Net /

0.00 g

451.64 g Brut  
RC

FOURNIER 1001744

Highly Confidential  
Subject to  
Protective Order2 - Pesée des bords de dissolution - Balance GALOGS

un litre de LSNa 0,1 M pèse 1004,0g -

12-10.07.07

042

13.06.97

09:18:40

Code

1

Code

3

Code

5

0.00 g

0.00 g

0.00 g

767.0 g Tare

698.8 g Tare

760.8 g Tare

0.00 g

0.00 g

0.00 g

1004.0 g Net

1004.0 g Net

1004.0 g Net

0.00 g

0.00 g

0.00 g

1771.0 g Brut

1702.8 g Brut

1764.9 g Brut

Code

2

Code

4

Code

6

0.00 g

0.00 g

0.00 g

768.8 g Tare

760.5 g Tare

753.0 g Tare

0.00 g

0.00 g

0.00 g

1004.0 g Net

1004.0 g Net

1004.0 g Net

0.00 g

0.00 g

0.00 g

1772.8 g Brut  
RC1764.5 g Brut  
RC1757.0 g Brut  
RC3. Conditions de dissolution.Dissolution <sup>est</sup> GAL 091 - Solax AT7.Température:  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  RC 13.06.97 AG 13/6/97

Vitesse: 90 TPM - RC 13.06.97 AG 13/6/97

4. Pesée des comprimés - Balance GAL 205.LF1787ER  
13.06.97 10:17:12  
Code lot C0200

0.0 mg

713.8 mg

0.0 mg

699.8 mg

0.0 mg

711.2 mg

0.0 mg

712.9 mg

0.0 mg

715.1 mg

0.0 mg

712.7 mg

RC

FOURNIER 1001745

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

5. lectures  
 sur spectrophotomètre UVIKON 922-GAL233 en cuve de 2mm<sup>043</sup>  
 de trajet optique à 290 nm.

LONGUEUR D ONDE FIXE 290 NM 2MM LSNA 0.1M 90TPM LOT C0200 LF178TER /

06-13-1997 11:33

Lambda	No.	Valeur_E
290.0	1	-0.0001_1 <i>Ain / Ain</i>
290.0	2	0.0033_1 <i>LSNa / LSNa</i>
290.0	3	0.0038_1
290.0	4	0.0038_1
290.0	5	0.0042_1 <i>0 mm</i>
290.0	6	0.0039_1
290.0	7	0.0043_1
290.0	8	0.0042_1
290.0	9	1.3308_1
290.0	10	1.2877_1
290.0	11	1.2621_1 <i>10 mm</i>
290.0	12	1.3332_1
290.0	13	1.1060_1
290.0	14	1.3109_1
290.0	15	1.4316_1
290.0	16	1.3964_1
290.0	17	1.4154_1 <i>16 mm</i>
290.0	18	1.4221_1
290.0	19	1.4225_1
290.0	20	1.4144_1
290.0	21	1.4438_1
290.0	22	1.4224_1
290.0	23	1.4315_1 <i>20 mm</i>
290.0	24	1.4367_1
290.0	25	1.4497_1
290.0	26	1.4310_1
290.0	27	1.4424_1
290.0	28	1.4169_1
290.0	29	1.4344_1 <i>20 mm</i>
290.0	30	1.4411_1
290.0	31	1.4619_1
290.0	32	1.4350_1

KONTRON INSTRUMENTS UVIKON 922

Opérateur *RC*.....

FOURNIER 1001746

Highly Confidential  
 Subject to  
 Protective Order

*M 10.07.97*

# DISSOLUTION

m:\commun\qlnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 13/06/97  
APPAREIL : GAL091GAL233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 20mm

TITRE : LF178TER comprimés lot C0200 - 0,1M 90 tpm  
N° CAHIER : LF178ter dissolution n°2 page 041  
FICHER : m:\commun\q\nq\donnbase\lf178ter\dissolution\lot C0200 0,1M 90tpm  
ELUANT : LNa 0,1M  
AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160	✓ en mg
dosage théorique	160	

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001747

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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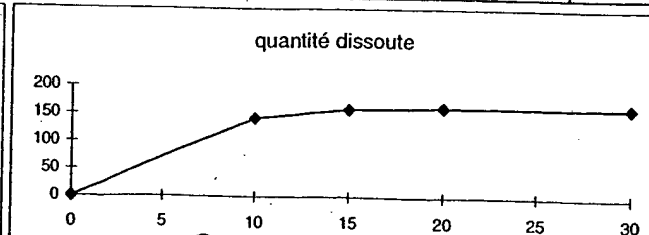
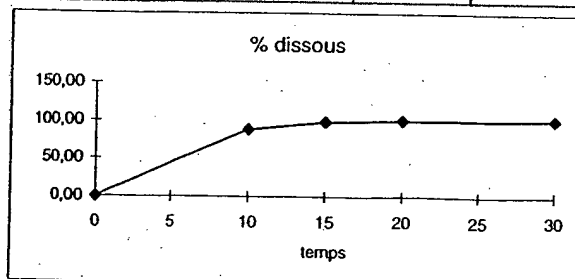
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOUTE

[illegible]



Parallèlement utilisation du 7<sup>ème</sup> bel pour des mesures avec ou sans filtration Miller HA ref-SLHA02S NB de 0,45 µm. Prélèvement 10 ml dont 5 ml filtré immédiatement et 5 ml restant dans la seringue et lecture après 20 min de temps de latence minimum -

Code

7

0.00 g

13.06.97

LF178TER

10:20:15

Code

lot 0200

675.9 g tare

0.0 mg

0.00 g

714.2 mg

1004.0 g Net -

0.00 g

1679.9 g Brut

RC

RC

LONGUEUR D ONDE FIXE 290 NM 2MM LSNA 0.1M 90TPM LOT C0200 LF178TER AVEC

06-13-1997 11:42

Lambda	No.	Valeur_E
290.0	1	0.0004_1 Air/Air
290.0	2	0.0035_1 LSNa/LSNa
290.0	3	0.0041_1 0 min
290.0	4	1.3458_1 10 min
290.0	5	1.4177_1 15 min
290.0	6	1.4188_1 20 min
290.0	7	1.4137_1 30 min

KONTRON INSTRUMENTS UVIKON 922

Opérateur .....

RC

LONGUEUR D ONDE FIXE 290 NM 2MM LSNA 0.1M 90TPM LOT C0200 LF178TER SANS

06-13-1997 11:47

Lambda	No.	Valeur_E
290.0	1	0.0000_1 Air/Air
290.0	2	0.0038_1 LSNa/LSNa
290.0	3	0.0045_1 0 min
290.0	4	1.3808_1 10 min
290.0	5	1.4344_1 15 min
290.0	6	1.4348_1 20 min
290.0	7	1.4343_1 30 min

FOURNIER 1001748

Highly Confidential  
Subject to  
Protective Order

RC 10.07.97

KONTRON INSTRUMENTS UVIKON 922

Opérateur .....

RC

M 22-07-07

FOURNIER 1001882

Highly Confidential  
Subject to  
Protective Order

M 30.07.97

FOURNIER 1001883

Highly Confidential  
Subject to  
Protective Order

Page	Date	Dénomination	178
103	03.07.97	Dissolution - lot 2395/01 RG - 14 kg L <sub>3</sub> Na 0,05N - 120 TPN	
106	06.07.97	Dissolution - lot RG 2395/01 - 14 kg. L <sub>3</sub> Na 0,05N - 120 TPN	
110	09.07.97	Dissolution - lot RG 2395/01 - 14 kg. L <sub>3</sub> Na 0,025N - 75 TPN	
114	18.07.97	Dissolution lot C $\phi$ 197 - L <sub>3</sub> Na 0,025N - 50 TPN	
118	18.07.97	" " - L <sub>3</sub> Na 0,025N - 80 TPN	
121	21.07.97	" " - L <sub>3</sub> Na 0,025N - 120 TPN	
124	21.07.97	" RG 2395/01 - 14 kg. L <sub>3</sub> Na 0,025N - 50 TPN	
128	21.07.97	" lot C $\phi$ 197 L <sub>3</sub> Na 0,05N - 50 TPN	
132	22.07.97	" " L <sub>3</sub> Na 0,05N - 75 TPN	
135	22.07.97	" lot C $\phi$ 197 L <sub>3</sub> Na 0,05N - 80 TPN	
139	23.07.97	" lot RG 2395/01 - 14 kg L <sub>3</sub> Na 0,05N - 50 TPN	
142	23.07.97	" Gelule Lipidil lot 43 Canada L <sub>3</sub> Na 0,1 - 80 TPN	
146	24.07.97	" L <sub>3</sub> 178ter lot C $\phi$ 197 L <sub>3</sub> Na 0,1N - 50 TPN	
149	24.07.97	Dissolution lot C $\phi$ 197 L <sub>3</sub> Na 0,1N - 75 TPN	
153	26.07.97	Dissolution lot C $\phi$ 197 L <sub>3</sub> Na 0,1N - 120 TPN	
156	26.07.97	Dissolution RG 2395/01 - 14 kg. L <sub>3</sub> Na 0,1N - 50 TPN	
159	28.07.97	Dissolution RG 2395/01 - 14 kg. L <sub>3</sub> Na 0,05N - 75 TPN	
163	28.07.97	Dissolution RG 2395/01 - 14 kg. L <sub>3</sub> Na 0,05N - 90 TPN	
166	29.07.97	Dissolution lot TG 192 - 18 kg. L <sub>3</sub> Na 0,025N - 75 TPN	
170	30.07.97	Dissolution RG 2395/01 - 14 kg. L <sub>3</sub> Na 0,025N - 120 TPN	

FOURNIER 1001884

Highly Confidential  
Subject to  
Protective Order

PAGE	DATE	SUJET
002	15.05.97	D3801 Gélules Lipidil 200 - lot 37 Canada - 0,025N / 75 TPM
008	16.05.97	Gélules Lipidil 200 - lot 39 Canada - 0,025N / 75 TPM
011	16.05.97	Gélules Lipidil 200 - lot 40 Canada - 0,025N / 75 TPM
014	03.06.97	LF 178 ter Canada - lot C0194 - 0,025N / 75 TPM
018	03.06.97	LF 178 ter Canada - lot C0197 - 0,025N / 75 TPM
021	05.06.97	LF 178 ter Canada - lot C0200 - 0,025N / 75 TPM
023	08.06.97	LF 178 ter - PK - lot 158 - 0,025N / 75 TPM
029	10.06.97	LF 178 ter - PK - lot 158 - 0,05N / 120 TPM
033	11.06.97	Dissolution - lot C0194 - 0,1M - 90 TPM
037	12.06.97	Dissolution - lot C0197 - 0,1M - 90 TPM
041	12.06.97	Dissolution - lot C0200 - 0,1M - 90 TPM
045	13.06.97	idem p041 - pot m <sup>st</sup> avec et sans microfiltration
048	17.06.97	Dissolution - lot 158 - 0,1M - 90 TPM
052	17.06.97	Dissolution - lot 234 - 0,1M - 90 TPM
055	18.06.97	Dissolution - lot 234 - 0,05M - 120 TPM
059	18.06.97	Dissolution - lot C0194 - 0,05M - 120 TPM
062	19.06.97	Dissolution - lot C0197 - 0,05M - 120 TPM
066	19.06.97	Dissolution - lot C0200 - 0,05M - 120 TPM
069	20.06.97	Dissolution - lot 234 - 0,025M - 75 TPM
073	24.06.97	Dissolution - lot 158 - 2 comprimés - 0,025M - 75 TPM
077	24.06.97	Dissolution - lot 2398/01 RG - 14Kg - 0,05M - 120 TPM
081	25.06.97	Dissolution - lot 2401/01 RG - 20Kg - 0,05M - 120 TPM
084	27.06.97	Dissolution - lot 2401/01 RG - 15Kg - 0,05M - 120 TPM
088	30.06.97	Dissolution - lot 2398/01 RG 14Kg - 0,05 <sup>re</sup> 0,1M - 90 TPM
092	01.07.97	Dissolution - lot 2401/01 RG - 15Kg - 0,1M - 90 TPM
095	01.07.97	Dissolution - lot 2401/01 RG - 20Kg - 0,1M - 90 TPM
099	03.07.97	Dissolution - lot 2395/01 RG - 14Kg - 0,1M - 90 TPM

FOURNIER 1001835  
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Subject to  
Protective Order

... Maitre Patrick NOURISSAT, notaire associé  
de la Société Civile Professionnelle " Patrick NOURISSAT, Didier  
NOURISSAT et Hugues MISSEY " titulaire d'un office Notarial  
dont le siège est à DUON, 25 rue Buffon.

CERTIFIE ET ATTESTE avoir procédé ce jour à la  
clôture du présent registre contenant 119 pages,  
l'avoir coté et paraphé en dernière page, destiné à  
l'enregistrement des formulations de médicaments pour  
les Laboratoires FOURNIER, centre de Biogalénique,  
42 rue de Longvic à Chenôve.

FAIT POUR SERVIR ET VALOIR CE QUE DE DROIT  
A DUON  
L'an DEUX MILLE 1994  
LE 10 Juin



FOURNIER 1001886  
Highly Confidential  
Subject to  
Protective Order

# DISSOLUTION

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 13/06/97  
APPAREIL : GAL091GAL233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 20mm

TITRE : LF178TER comprimés lot C0200 - 0,1M 90 tpm  
N° CAHIER : LF178ter dissolution n°2 page 045  
FICHIER : m:\commun\glnq\donnbases\lf178ter\dissolution\lot C0200 0,1M 90tpm avec filtre  
ELUANT : LSNa 0,1M  
AGITATION : 90 TPM AVEC FILTRATION

m:\commun\glnq\traidon\distem5  
date édition: le 06/02/97

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00					
quantité de principe actif	160,00	0,00	0,00	0,00	0,00	0,00

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	10
	volume en ml	CELLULE 1
0	0	0
10	1000	1,346
15	1000	1,418
20	1000	1,419
30	1000	1,414

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1
0,0	0,00	0
10,0	93,47	93,47
15,0	99,41	99,41
20,0	100,46	100,46
30,0	101,10	101,10
0,0		
0,0		
0,0		
0,0		
0,0		
0,0		
0,0		
0,0		

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1
0,0	0	0
10,0	149,56	149,56
15,0	159,05	159,05
20,0	160,74	160,74
30,0	161,76	161,76

Temps      Ecart entre les 3 mesures en %  
10 min      2,43%      Diss.  
15 min      1,13%  
20 min      1,15%  
30 min      1,43%

FOURNIER 1001749

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

047

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 13/06/97  
APPAREIL : GAL091GAL233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 20mm

TITRE : LF178TER comprimés lot C0200 - 0,1M 90 tpm  
N° CAHIER : LF178ter dissolution n°2 page 045  
FICHIER : m:\commun\glnq\donnbase\LF178ter\dissolution\lot C0200 0,1M 90tpm sans filtre  
ELUANT : LSNa 0,1M  
AGITATION : 90 TPM AVEC FILTRATION

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00					
quantité de principe actif	160,00	0,00	0,00	0,00	0,00	0,00

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	10
0	0	0
10	1000	1,381
15	1000	1,434
20	1000	1,435
30	1000	1,434

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1
0,0	0,00	0
10,0	95,90	95,90
15,0	100,54	100,54
20,0	101,61	101,61
30,0	102,53	102,53
0,0		
0,0		
0,0		
0,0		
0,0		
0,0		
0,0		
0,0		

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1
0,0	0	0
10,0	153,44	153,44
15,0	160,87	160,87
20,0	162,57	162,57
30,0	164,06	164,06

Etant donné l'écart entre les deux mesures, il serait préférable d'effectuer la filtration Miller 945µm juste après le prélèvement et non pas avant la lecture.  
Dans tout les cas, il faut adapter la filtration par rapport aux temps de prélèvements.

1<sup>er</sup> cas: lecture à temps 20, 40, 60, 120 min  
prélever, mettre un filtre Miller 945µm  
faire la lecture, l'intervalle de

temps entre deux lectures est suffisant pour faire les lectures après prélèvement.  
2<sup>nd</sup> cas- lecture à temps (5, 10, 15, 20, 30 min, prélever, mettre un filtre Miller 945µm, filtrer dans un tube à essai, en attente de la lecture.

FOURNIER 1001750

Highly Confidential  
Subject to  
Protective Order

M 10.07.97



17.06.97 048

Dissolution de comprimés LF178TER  
lot 158 - 100 mg  
LSNa 0,1 M - 90 TPM

# 1. Préparation du milieu de dissolution - Balance GAL065.

17.06.97 Eau purif 10:48:22  
0.00 g

642.4 g Tare

0.00 g

2671.0 g Net

0.00 g

3313.4 g Brut

0.00 g

645.0 g Tare

0.00 g

2684.7 g Net

0.00 g

3329.8 g Brut

0.00 g

644.5 g Tare

0.00 g

2582.5 g Net

0.00 g

3226.9 g Brut

0.00 g

644.8 g Tare

0.00 g

2420.6 g Net

0.00 g

3065.4 g Brut

0.00 g

644.7 g Tare

0.00 g

3038.8 g Net

0.00 g

3683.5 g Brut

RC

Pesée de LSNa pour un  
milieu à 0,1 M -  $P_{\text{compr}} = 288,4 \text{ g}$   
 $13397,6 \times 0,1 \times 288,4 = 386,39 \text{ g}$   
1000

Total de l'eau purifiée  
pesées

2671,0 g

+ 2684,7 g

+ 2582,5 g

+ 2420,6 g

+ 3038,8 g

13397,6 g

17.06.97 LSNa 11:14:49  
Code APP 1547

0.00 g

143.24 g Tare

0.00 g

386.39 g Net

0.00 g

529.61 g Brut  
RC

# 2. Pesée des bords de dissolution - Balance GAL065.

1 litre de LSNa 0,1 M pèse 0,1 M 1004,0 g

FOURNIER 1001751  
Highly Confidential  
Subject to  
Protective Order

12.10.07.97

17.06.97  
Code

13:03:09  
1

Code

3

Code

5

049

0.00 g

760.8 g tare

0.00 g

1004.0 g Net

0.00 g

1764.8 g Brut

0.00 g

760.5 g tare

0.00 g

1004.0 g Net

0.00 g

1764.5 g Brut

0.00 g

768.8 g tare

0.00 g

1004.0 g Net

0.00 g

1772.8 g Brut

Code

2

Code

4

Code

6

0.00 g

767.0 g tare

0.00 g

1004.0 g Net

0.00 g

1771.0 g Brut

0.00 g

753.0 g tare

0.00 g

1004.0 g Net

0.00 g

1757.0 g Brut

0.00 g

698.8 g tare

0.00 g

1004.0 g Net

0.00 g

1702.8 g Brut

### 3 - Conditions de dissolution

Dissolvant GAL 091 - Solax AT 7

Température:  $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  RC 17.06.97 RC 17/6/97

Vitesse: 90 TPM RC 17.06.97 RC 17/6/97

### 4 - Pesée des comprimés - Balance GAL 205

17.06.97 14:49:11

Code LAFSTER 158

0.0 mg

559.9 mg

0.0 mg

561.7 mg

0.1 mg

0.0 mg

552.0 mg

0.0 mg

556.4 mg

0.0 mg

554.6 mg

0.0 mg

561.8 mg

RC

FOURNIER 1001752  
Highly Confidential  
Subject to  
Protective Order

M 10.07.97

050

5-lectures sur spectrophotomètre UVIKON 922 GAL233 en cuve de 2mm  
à 290nm

LONGUEUR D ONDE FIXE 290 NM 2 MM LF178TER LOT. 158 LSNA 0.1M 90TPM /

06-17-1997 16:16

Lambda	No.	Valeur_E
290.0	1	-0.0004_1 <i>Au/Au</i>
290.0	2	0.0038_1 <i>18Na/LSNa</i>
290.0	3	0.0040_1
290.0	4	0.0038_1
290.0	5	0.0046_1 <i>0mm</i>
290.0	6	0.0037_1
290.0	7	0.0034_1
290.0	8	0.0035_1
290.0	9	0.8628_1
290.0	10	0.8819_1
290.0	11	0.8714_1 <i>10mm</i>
290.0	12	0.8848_1
290.0	13	0.8811_1
290.0	14	0.9027_1
290.0	15	0.8995_1
290.0	16	0.9201_1
290.0	17	0.8998_1 <i>15mm</i>
290.0	18	0.9025_1
290.0	19	0.9105_1
290.0	20	0.9427_1
290.0	21	0.9091_1
290.0	22	0.9291_1
290.0	23	0.8763_1 <i>20mm</i>
290.0	24	0.8812_1
290.0	25	0.8873_1
290.0	26	0.9513_1
290.0	27	0.9176_1
290.0	28	0.9331_1
290.0	29	0.9109_1 <i>25mm</i>
290.0	30	0.9149_1
290.0	31	0.9216_1
290.0	32	0.9565_1

KONTRON INSTRUMENTS UVIKON 922

Opérateur *BC*

FOURNIER 1001753

Highly Confidential  
Subject to  
Protective Order

*M 10.07.97*

051

OPERATEUR : ROSSELIN C  
DATE : 17/06/97  
APPAREIL : GAL091 GAL233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2 mm

TITRE : LF178TER comprimés lot 158 - 0,1M 90 tpm  
N° CAHIER : LF178ter dissolution n°2 page048 /  
FICHIER : m:\commun\glnq\lf178ter\dissolution\lot PK 158 0,1M 90 tpm /  
ELUANT : LSNa 0,1M /  
AGITATION : 90 TPM /

masse théorique	100	
dosage théorique	100	en mg

FOURNIER 1001754

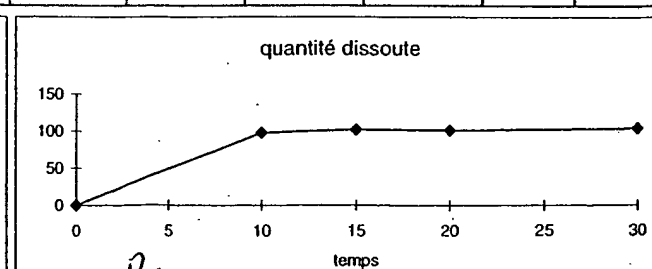
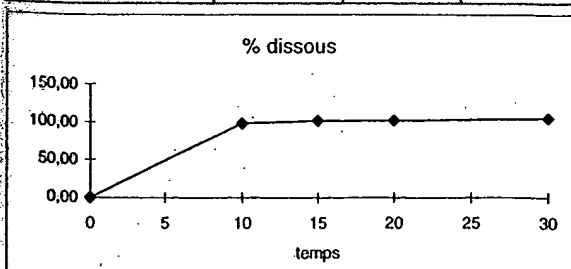
masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
100,00	100,00	100,00	100,00	100,00	100,00
100,00	100,00	100,00	100,00	100,00	100,00

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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volume prélevé en ml 5

[illegible][illegible][illegible]

M 10.07.97

Re

17.06.97

Dissolution de comprimés LF 178TER  
lot 234 - 100mg  
LSNa g1M - 90 TPM

1 - Préparation du milieu de dissolution.

cf page 048.

2. Pesée des bés de dissolution. Balance GAL 065

1 litre de LSNa g1M pèse 1004,0g -

17.06.97

17:13:04

Code

1

Code

3

Code

5

0.00 g

0.00 g

0.00 g

768.8 g Tare

767.0 g Tare

698.8 g Tare

0.00 g

0.00 g

0.00 g

1004.0 g Net -

1004.0 g Net -

1004.0 g Net -

0.00 g

0.00 g

0.00 g

1772.8 g Bunk

1771.0 g Bunk

1702.8 g Bunk

Code

2

Code

4

Code

6

0.00 g

0.00 g

0.00 g

753.0 g Tare

760.8 g Tare

760.5 g Tare

0.00 g

0.00 g

0.00 g

1004.0 g Net -

1004.0 g Net -

1004.0 g Net -

0.00 g

0.00 g

0.00 g

1757.0 g Bunk RC

1764.9 g Bunk RC

1764.5 g Bunk RC

3. Conditions de dissolution

Dissolutes Sotax AT7 - GAL 091 -

Température:  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  RC 18.06.97 CC 18/06/97

Agitation: 90 TPM. RC 18.06.97 CC 18/06/97

4. Pesée des comprimés - Balance GAL 205 -

FOURNIER 1001755  
Highly Confidential  
Subject to  
Protective Order

M 10.07.97

LF178TER

18.06.97 09:58:11

Code

lot 234

0.0000 g  
0.5588 g  
0.0000 g  
0.5732 g  
0.0000 g  
0.5662 g  
0.0000 g  
0.5607 g  
0.0000 g  
0.5643 g  
0.0000 g  
0.5676 g

RL

## 5 - lectures

sur spectrophotomètre UVIKON 922 GAL 233 en cuve de  
2 mm de trajet optique à 290 nm.

LONGUEUR D ONDE FIXE 290NM 2 MM LF178TER LOT 234 LSNA 0.1M 90TPM /

06-18-1997 11:23

Lambda	No.	Valeur_E
290.0	1	0.0001_1 <i>Ai/Ai</i>
290.0	2	-0.0033_1 <i>LSNa/LSNa</i>
290.0	3	-0.0046_1
290.0	4	0.0012_1
290.0	5	-0.0012_1 <i>0 min</i>
290.0	6	0.0014_1
290.0	7	-0.0015_1
290.0	8	-0.0015_1
290.0	9	0.7767_1
290.0	10	0.8145_1
290.0	11	0.7870_1 <i>10 min</i>
290.0	12	0.7982_1
290.0	13	0.8043_1
290.0	14	0.8068_1
290.0	15	0.8602_1
290.0	16	0.8942_1
290.0	17	0.8933_1 <i>15 min</i>
290.0	18	0.8875_1
290.0	19	0.8729_1
290.0	20	0.9025_1
290.0	21	0.8858_1
290.0	22	0.9157_1
290.0	23	0.9200_1 <i>20 min</i>
290.0	24	0.9001_1
290.0	25	0.8965_1
290.0	26	0.9338_1
290.0	27	0.9042_1
290.0	28	0.9371_1
290.0	29	0.9317_1 <i>30 min</i>
290.0	30	0.9163_1
290.0	31	0.9116_1
290.0	32	0.9453_1

FOURNIER 1001756

Highly Confidential  
Subject to  
Protective Order

CONTRON INSTRUMENTS UVIKON 922

Opérateur .....

m 10.07.97

RL

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 18/06/97 ✓  
APPAREIL : GAL091 GAL233 ✓  
LONGUEUR D'ONDE : 290 nm ✓  
CUVE en mm: 2 mm ✓

TITRE : LF178TER comprimés PK lot 234 - 0,1M 90tpm  
N° CAHIER : LI178TER dissolution n°2 page 052  
FICHIER : m:\commun\gmg\LF178ter\dissolution\lot PK 234 0,1M 90tpm  
ELUANT : LSNa 0,1M  
AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	100	
dosage théorique	100	en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	100,00	100,00	100,00	100,00	100,00	100,00
quantité de principe actif	100,00	100,00	100,00	100,00	100,00	100,00

FOURNIER 1001757

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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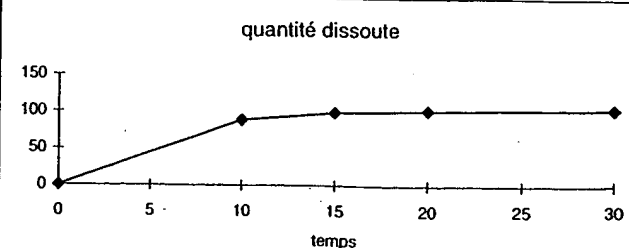
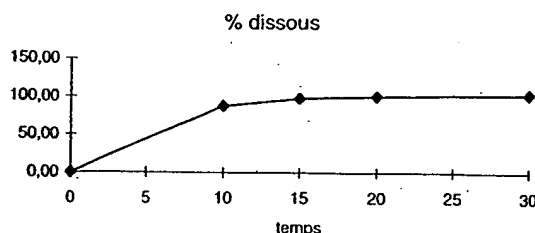
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOUTE

[illegible]

m. 10.07.47 RC

18.06.97

Dissolution LF 178 TER comprimés

055

lot 234 - 100 mg

LSNa 0,05 H - 120 TPM

## 1 - Préparation du milieu de dissolution - Balance GAL065.

18.06.97 Eau purifiée 1:56:55

0.00 g

0.00 g

642.4 g Tare

644.5 g Tare

0.00 g

0.00 g

2852.9 g Net

2742.5 g Net

0.00 g

0.00 g

3495.3 g Brut

3387.0 g Brut

0.00 g

0.00 g

644.4 g Tare

644.3 g Tare

0.00 g

0.00 g

2946.5 g Net

2885.8 g Net

0.00 g

0.00 g

3590.9 g Brut

3530.1 g Brut

0.00 g

RC

644.4 g Tare

0.00 g

2932.8 g Net

0.00 g

3577.2 g Brut  
RC

- Pesée de LSNa pour un  
milieu 0,05 H  $M_{LSNa} = 288,4 g$   
 $\frac{14360,5 \times 0,05 \times 288,4}{1000} = 207,08 g$

Total de l'eau purifiée  
pesée.

2852,9 g

+ 2946,5 g

+ 2932,8 g

+ 2742,5 g

+ 2885,8 g

14360,5 g

18.06.97 LSNa 12:29:58

Code Am 1547

0.00 g

143.23 g

0.00 g

207.09 g

0.00 g

350.32 g

## 2 - Pesée des lots de dissolution - Balance GAL065.

1 litre de LSNa 0,05 H pèse 1002,0 g.

M. 10.07.97

FOURNIER 1001758

Highly Confidential  
Subject to  
Protective Order



0.00 g

760.5 g Tare

0.00 g

1002.0 g Net

0.00 g

1762.5 g Bunk

Code

2

0.00 g

753.0 g Tare

0.00 g

1002.0 g Net

0.00 g

1755.0 g Bunk  
AC

0.00 g

760.9 g Tare

0.00 g

1002.0 g Net

0.00 g

1762.9 g Bunk

Code

4

0.00 g

768.8 g Tare

0.00 g

1002.0 g Net

0.00 g

1770.8 g Bunk  
AC

0.00 g

767.0 g Tare

0.00 g

1002.0 g Net

0.00 g

1769.0 g Bunk

Code

6

0.00 g

698.8 g Tare

0.00 g

1002.0 g Net

0.00 g

1700.8 g Bunk  
AC

### 3. Conditions de dissolution

Dissolvent Solax AT7 GAL091

Température :  $27^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  AC 18.06.97 CC 18/06/97

Agitation : 120TPM AC 18.06.97 CC 18/06/97

### 4. Pesée des comprimés - Balance GAL205

18.06.97 14:41:22

18.06.97 14:41:48

Code 1807ER Lot 234

0.0 mg

559.6 mg

0.0 mg

558.7 mg

0.0 mg

564.6 mg

0.0 mg

552.5 mg

0.1 mg

0.0 mg

559.1 mg

0.0 mg

555.5 mg

AC

### 5. lectures

Sur Spectrophotométrie UVIKON 922 GAL233 en cuve  
de 2mm à 290 nm

FOURNIER 1001759

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

057

LONGUEUR D ONDE FIXE 290NM 2 MM LF178TER LOT 234 LSNA 0.05M 120TPM /

06-18-1997 16:40

Lambda	No.	Valeur_E
290.0	1	-0.0001_1
290.0	2	-0.0023_1
290.0	3	-0.0037_1
290.0	4	-0.0038_1
290.0	5	-0.0046_1
290.0	6	-0.0045_1
290.0	7	-0.0042_1
290.0	8	-0.0047_1
290.0	9	0.7534_1
290.0	10	0.7357_1
290.0	11	0.7461_1
290.0	12	0.7449_1
290.0	13	0.7617_1
290.0	14	0.7690_1
290.0	15	0.8201_1
290.0	16	0.8139_1
290.0	17	0.8487_1
290.0	18	0.8055_1
290.0	19	0.8316_1
290.0	20	0.8336_1
290.0	21	0.8621_1
290.0	22	0.8461_1
290.0	23	0.8850_1
290.0	24	0.8344_1
290.0	25	0.8633_1
290.0	26	0.8692_1
290.0	27	0.8910_1
290.0	28	0.8757_1
290.0	29	0.9218_1
290.0	30	0.8550_1
290.0	31	0.8866_1
290.0	32	0.8967_1
290.0	33	0.9037_1
290.0	34	0.8891_1
290.0	35	0.9247_1
290.0	36	0.8728_1
290.0	37	0.8961_1
290.0	38	0.9039_1
290.0	39	0.9022_1
290.0	40	0.8920_1
290.0	41	0.9263_1
290.0	42	0.8732_1
290.0	43	0.8991_1
290.0	44	0.9070_1
290.0	45	0.9061_1
290.0	46	0.8939_1
290.0	47	0.9262_1
290.0	48	0.8747_1
290.0	49	0.8958_1
290.0	50	0.9053_1

$A_n / A_n$   
 $LSNa / LSNa$

0 min

10 min

15 min

20 min

30 min

40 min

50 min

60 min

FOURNIER 1001760

Highly Confidential  
 Subject to  
 Protective Order

KONTRON INSTRUMENTS UVIKON 922

Opérateur .....

AC

M 10.07.97

058

**DISSOLUTION**

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

**CONDITIONS ET MODES OPERATOIRES**

OPERATEUR : ROSSELIN C  
 DATE : 18/06/97  
 APPAREIL : GAL091 GAL233  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2 mm

TITRE : LF178TER comprimés PK lot 234 - 0,05M 120tpm  
 N° CAHIER : LI178TER dissolution n°2 page055  
 FICHER : m:\commun\glnq\lf178ter\dissolution\lot PK 234 0,05M 120tpm  
 ELUANT : LSNa 0,05M  
 AGITATION : 120 TPM

**PREPARATION DES ECHANTILLONS**

masse théorique 100  
 dosage théorique 100 en mg

masse de la prise d'essai  
 quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
100,00	100,00	100,00	100,00	100,00	100,00
100,00	100,00	100,00	100,00	100,00	100,00

**FOURNIER 1001761**

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Témoin 100mg/l 0,900

**SUIVI DE LA DISSOLUTION**

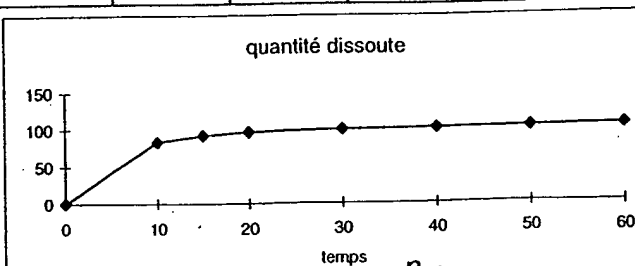
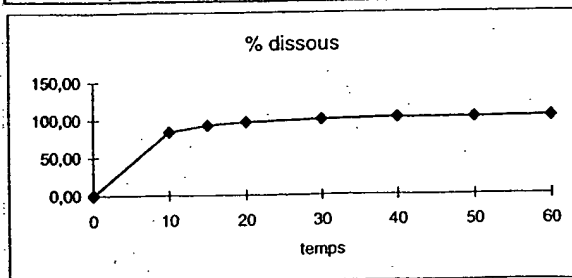
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,753	0,736	0,746	0,745	0,762	0,769
15	1000	0,82	0,814	0,849	0,806	0,832	0,834
20	1000	0,862	0,846	0,885	0,834	0,863	0,869
30	1000	0,891	0,876	0,922	0,855	0,887	0,897
40	1000	0,904	0,889	0,925	0,873	0,896	0,904
50	1000	0,902	0,892	0,926	0,873	0,899	0,907
60	1000	0,906	0,894	0,926	0,875	0,896	0,905

**RESULTATS EN % DISSOUS**

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	83,54	83,67	81,78	82,89	82,78	84,67	85,44	1,34	1,61
15,0	92,18	91,53	90,85	94,75	89,97	92,87	93,09	1,73	1,88
20,0	96,41	96,65	94,86	99,22	93,53	96,77	97,45	1,99	2,07
30,0	100,02	100,35	98,66	103,82	96,33	99,92	101,04	2,49	2,49
40,0	101,68	102,29	100,60	104,67	98,80	101,41	102,32	1,96	1,93
50,0	102,33	102,57	101,42	105,29	99,29	102,24	103,15	1,98	1,94
60,0	102,88	103,52	102,14	105,81	99,99	102,41	103,43	1,92	1,86
0,0									
0,0									
0,0									
0,0									
0,0									

**RESULTATS EN QUANTITE DISSOUTE**

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	83,54	83,67	81,78	82,89	82,78	84,67	85,44	1,34	1,61
15,0	92,18	91,53	90,85	94,75	89,97	92,87	93,09	1,73	1,88
20,0	96,41	96,65	94,86	99,22	93,53	96,77	97,45	1,99	2,07
30,0	100,02	100,35	98,66	103,82	96,33	99,92	101,04	2,49	2,49
40,0	101,68	102,29	100,60	104,67	98,80	101,41	102,32	1,96	1,93
50,0	102,33	102,57	101,42	105,29	99,29	102,24	103,15	1,98	1,94
60,0	102,88	103,52	102,14	105,81	99,99	102,41	103,43	1,92	1,86
0,0									
0,0									
0,0									
0,0									
0,0									



PL

m. 10.07.97

18.06.97

Dissolution comprimés LF178TER

059

lot CO 194 - 160 mg

LSNa 0,05 M - 120 TPM

### 1 - Préparation du milieu de dissolution.

Cf page 055

### 2 - Pesée des bords de dissolution - Balance GALL205<sup>RC</sup>066

1 litre de LSNa 0,05 M pèse 1002,0g

18.06.97 18:04:27

Code Eau purifiée 1

Code 3

0.00 g

0.00 g

Code 5

767.0 g Tare

760.8 g Tare

0.00 g

0.00 g

~~0.01~~ g RC

760.5 g Tare

1002.0 g Net -

0.00 g

0.00 g

0.00 g

1002.0 g Net -

1002.0 g Net -

1769.0 g Bulk

0.00 g

0.00 g

Code 2

1762.8 g Bulk

1762.4 g Bulk

0.00 g

Code 4

Code 6

768.8 g Tare

0.00 g

0.00 g

0.00 g

753.0 g Tare

698.8 g Tare

1002.0 g Net -

0.00 g

0.00 g

~~0.01~~ g RC

1002.0 g Net -

1002.0 g Net -

0.00 g

0.00 g

0.00 g

1770.8 g Bulk  
RC

1755.0 g Bulk  
RC

1700.8 g Bulk  
RC

### 3 - Conditions de de dissolution

Dissolubilité Solax AT7 GAL091

Température :  $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  RC 19.06.97 19.06.97

Agitation : 120 TPM RC 18<sup>RC</sup>.06.97 19.06.97

FOURNIER 1001762

Highly Confidential  
Subject to  
Protective Order

12 10.07.97

060

## 4. Pesée des comprimés - Balance GAL 205

19.05.97  
Code  
19.05.97  
Lot C0194  
LF178TER

0.0 mg  
708.8 mg  
0.0 mg  
712.9 mg  
0.0 mg  
711.6 mg  
0.0 mg  
706.7 mg  
0.0 mg  
709.7 mg  
0.0 mg  
702.3 mg

RL

## 5 - lectures

Suiv spectrophomètre UVIKON 922 GAL 233 en cuve de 2mm  
à 290 nm.

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER LOT C0194 LSNA 0.05M 120TPM

06-19-1997

Lambda No. Valeur\_E

290.0	1	0.0000	1	Ain / Ain
290.0	2	-0.0006	1	LSNa / LSNa
290.0	3	-0.0006	1	
290.0	4	-0.0004	1	
290.0	5	-0.0008	1	0mm
290.0	6	-0.0006	1	
290.0	7	-0.0003	1	
290.0	8	-0.0000	1	
290.0	9	1.2382	1	
290.0	10	1.2334	1	
290.0	11	1.2339	1	10mm
290.0	12	1.2435	1	
290.0	13	1.2521	1	
290.0	14	1.2148	1	
290.0	15	1.3745	1	
290.0	16	1.3662	1	
290.0	17	1.3838	1	15mm
290.0	18	1.3668	1	
290.0	19	1.3715	1	
290.0	20	1.3648	1	
290.0	21	1.4147	1	
290.0	22	1.4066	1	
290.0	23	1.4201	1	20mm
290.0	24	1.4023	1	
290.0	25	1.4167	1	
290.0	26	1.4111	1	
290.0	27	1.4352	1	
290.0	28	1.4280	1	
290.0	29	1.4474	1	30mm
290.0	30	1.4297	1	
290.0	31	1.4357	1	
290.0	32	1.4392	1	
290.0	33	1.4451	1	
290.0	34	1.4309	1	
290.0	35	1.4474	1	40mm
290.0	36	1.4290	1	
290.0	37	1.4377	1	
290.0	38	1.4361	1	
290.0	39	1.4337	1	
290.0	40	1.4229	1	
290.0	41	1.4489	1	50mm
290.0	42	1.4210	1	
290.0	43	1.4280	1	
290.0	44	1.4347	1	
290.0	45	1.4257	1	
290.0	46	1.4200	1	
290.0	47	1.4452	1	60mm
290.0	48	1.4156	1	
290.0	49	1.4251	1	
290.0	50	1.4338	1	

FOURNIER 1001763

Highly Confidential  
Subject to  
Protective Order

UVIKON 922

Opérateur

RL

m 10.07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

061

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 19/06/97  
APPAREIL : GAL091GAL233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2 mm

TITRE : LF178TER comprimés lot C0194 - 0,05M 120tpm  
N° CAHIER : LF178ter dissolution n°2 page 059  
FICHIER : m:\commun\glnq\lf178ter\dissolution\lot C0194 0,05M 120tpm  
ELUANT : LSNa 0,05M  
AGITATION : 120 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001764

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

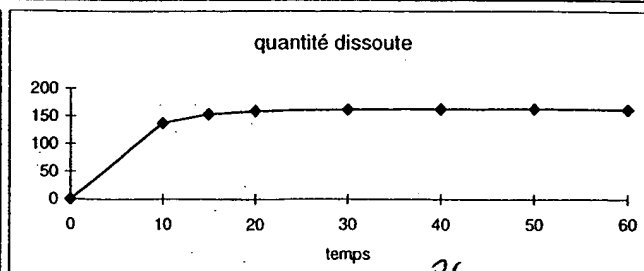
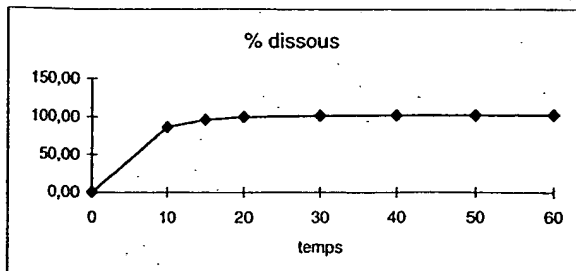
volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
10	1000	1,238	1,233	1,234	1,244	1,252	1,215
15	1000	1,375	1,366	1,384	1,367	1,372	1,365
20	1000	1,415	1,407	1,42	1,402	1,417	1,411
30	1000	1,435	1,428	1,447	1,43	1,436	1,439
40	1000	1,445	1,431	1,447	1,429	1,438	1,436
50	1000	1,434	1,423	1,449	1,421	1,428	1,435
60	1000	1,426	1,42	1,445	1,416	1,425	1,434

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	85,83	85,97	85,63	85,69	86,39	86,94	84,38	0,87	1,01
15,0	95,67	95,92	95,29	96,54	95,36	95,71	95,21	0,50	0,53
20,0	98,96	99,17	98,61	99,52	98,27	99,31	98,88	0,47	0,47
30,0	101,11	101,05	100,56	101,89	100,70	101,13	101,32	0,47	0,47
40,0	101,73	102,24	101,26	102,39	101,13	101,76	101,61	0,51	0,50
50,0	101,81	101,98	101,20	103,03	101,07	101,57	102,04	0,71	0,70
60,0	102,03	101,92	101,49	103,26	101,21	101,86	102,47	0,73	0,72
0,0									
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	137,33	137,56	137,00	137,11	138,22	139,11	135,00	1,39	1,01
15,0	153,08	153,47	152,46	154,46	152,58	153,14	152,34	0,81	0,53
20,0	158,34	158,67	157,78	159,23	157,23	158,90	158,21	0,75	0,47
30,0	161,77	161,68	160,89	163,02	161,12	161,80	162,11	0,76	0,47
40,0	162,77	163,59	162,02	163,83	161,80	162,82	162,57	0,82	0,50
50,0	162,90	163,17	161,93	164,85	161,71	162,51	163,26	1,14	0,70
60,0	163,25	163,08	162,38	165,21	161,94	162,97	163,95	1,18	0,72
0,0									
0,0									
0,0									
0,0									
0,0									



M 10.07.47

RC

062  
19.06.97

Dissolution comprimés LF178TER  
lot CO197 - 160 mg  
LSNa 9,05 M - 120 TPH

# 1. Préparation du milieu de dissolution - Balance GAL065

19.06.97 Jan 11:15:23

0.00 g purifiée

642.4 g Tare

0.00 g

2806.3 g Net

0.00 g

3448.6 g Bulk

0.00 g

645.0 g Tare

0.00 g

2809.5 g Net

0.00 g

3454.5 g Bulk

0.00 g

645.6 g Tare

0.00 g

2834.0 g Net

0.00 g

3479.6 g Bulk RC

0.00 g

644.7 g Tare

0.00 g

2858.2 g Net

0.00 g

3502.9 g Bulk

0.00 g

645.2 g Tare

0.00 g

2786.8 g Net

0.00 g

3432.1 g Bulk RC

Pesée de LSNa pour un

milieu à 9,05 M -  $PH_{LSNa} = 288,4g$

$\frac{14094,8 \times 9,05 \times 288,4}{1000} = 203,25g$

Total eau purifiée pesée

2806,3 g

+ 2809,5 g

+ 2834,0 g

+ 2858,2 g

+ 2786,8 g

14094,8 g

19.06.97 RC 11:40:23  
Code LSNa ARR1547

0.00 g

143.21 g Tare

0.00 g

203.25 g Net

0.00 g

346.50 g Bulk

## 2. Pesée des lots à dissolution

Balance GAL065

Un litre de LSNa 9,05 M pèse

1002,0g

FOURNIER 1001765

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Subject to  
Protective Order

M 10.07.97

19.06.97  
Code

10.06.97  
1

Code

3

Code

5

063

0.00 g  
768.8 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1770.8 g Bunk

0.00 g  
767.0 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1769.0 g Bunk

0.00 g  
760.5 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1762.5 g Bunk

Code

2

Code

4

Code

6

0.00 g  
698.8 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1700.8 g Bunk  
RC

0.00 g  
753.0 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1755.1 g Bunk  
RC

0.00 g  
760.8 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1762.9 g Bunk  
RC

### 3. Conditions de dissolution

Dissolutor Solax AT7 - GAL091

Température:  $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  19.06.97 AC 19/6/97

Agitation: 120T AC 19.06.97 AC 19/6/97.

### 4. Pesée des comprimés - Balance GAL205 -

19.06.97 17:18:14:50:48  
Code Lot 0197

0.0 mg  
713.8 mg  
~~0.1 mg~~ RC  
0.0 mg  
712.3 mg  
0.0 mg  
719.5 mg  
0.0 mg  
707.6 mg  
0.0 mg  
718.3 mg  
0.0 mg  
710.3 mg

RC

FOURNIER 1001766

Highly Confidential  
Subject to  
Protective Order

### 5. Lectures

Du spectrophotomètre UVIKON 922 - GAL233 - au cuve de 2 mm  
de trajet optique à 290 nm - M 10.07.97



LONGUEUR D ONDE FIXE 290NM 2MM LF178TER LOT C0197 LSNA 0.05M 120TPM /

06-19-1997 16:22

Lambda	No.	Valeur_E
290.0	1	0.0001_1 <i>Air / Air</i>
290.0	2	0.0013_1 <i>LSNA / LSNA</i>
290.0	3	0.0025_1
290.0	4	0.0001_1
290.0	5	-0.0001_1 <i>0 min</i>
290.0	6	-0.0000_1
290.0	7	0.0000_1
290.0	8	0.0001_1
290.0	9	1.2406_1
290.0	10	1.2518_1
290.0	11	1.2330_1 <i>10 min</i>
290.0	12	1.2653_1
290.0	13	1.2610_1
290.0	14	1.2660_1
290.0	15	1.3794_1
290.0	16	1.3712_1
290.0	17	1.3900_1 <i>15 min</i>
290.0	18	1.3756_1
290.0	19	1.3965_1
290.0	20	1.3741_1
290.0	21	1.4206_1
290.0	22	1.3977_1
290.0	23	1.4387_1 <i>20 min</i>
290.0	24	1.4057_1
290.0	25	1.4285_1
290.0	26	1.4088_1
290.0	27	1.4340_1
290.0	28	1.4101_1
290.0	29	1.4541_1 <i>30 min</i>
290.0	30	1.4289_1
290.0	31	1.4489_1
290.0	32	1.4219_1
290.0	33	1.4300_1
290.0	34	1.4103_1
290.0	35	1.4517_1 <i>40 min</i>
290.0	36	1.4198_1
290.0	37	1.4344_1
290.0	38	1.4144_1
290.0	39	1.4296_1
290.0	40	1.4097_1
290.0	41	1.4444_1 <i>50 min</i>
290.0	42	1.4123_1
290.0	43	1.4406_1
290.0	44	1.4172_1
290.0	45	1.4132_1
290.0	46	1.3931_1
290.0	47	1.4415_1 <i>60 min</i>
290.0	48	1.4062_1
290.0	49	1.4051_1
290.0	50	1.4371_1

FOURNIER 1001767  
Highly Confidential  
Subject to  
Protective Order

# DISSOLUTION

m:\commun\glnq\traitdon\distern5

date édition: le 06/02/97

065

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
 DATE : 19/06/97  
 APPAREIL : GAL091GAL233  
 LONGUEUR D'ONDE : 290 nm  
 CUVES en mm : 2 mm

TITRE : LF178TER comprimés lot C0197 - 0,05M 120tpm  
 N° CAHIER : LF178ter dissolution n°2 page 062  
 FICHER : m:\commun\glnq\lf178ter\dissolution\lot C0197 0,05M 120tpm  
 ELUANT : LSNa 0,05M  
 AGITATION : 120 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/l 0,900

FOURNIER 1001768

Highly Confidential  
 Subject to  
 Protective Order

## SUIVI DE LA DISSOLUTION

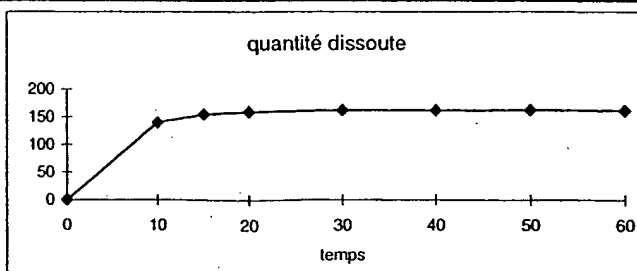
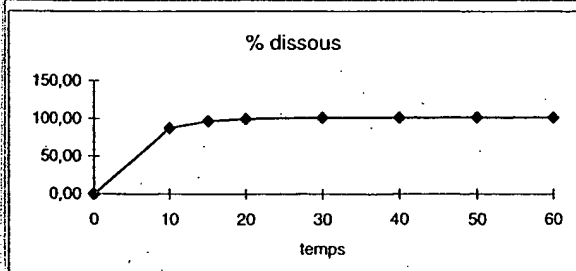
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,241	1,252	1,233	1,265	1,261	1,266
15	1000	1,379	1,371	1,39	1,376	1,397	1,374
20	1000	1,421	1,398	1,439	1,406	1,429	1,409
30	1000	1,434	1,41	1,454	1,429	1,449	1,422
40	1000	1,43	1,41	1,452	1,42	1,434	1,414
50	1000	1,43	1,41	1,444	1,412	1,441	1,417
60	1000	1,413	1,393	1,442	1,406	1,405	1,437

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	87,01	86,18	86,94	85,63	87,85	87,57	87,92	0,94	1,08
15,0	96,35	96,19	95,64	96,96	95,99	97,45	95,86	0,70	0,73
20,0	99,32	99,59	97,99	100,84	98,56	100,16	98,76	1,07	1,08
30,0	100,92	100,99	99,31	102,38	100,64	102,04	100,16	1,15	1,14
40,0	100,98	101,21	99,80	102,75	100,51	101,51	100,09	1,08	1,07
50,0	101,40	101,70	100,29	102,70	100,45	102,49	100,79	1,04	1,03
60,0	101,23	101,02	99,60	103,06	100,52	100,49	102,67	1,35	1,34
0,0									
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	139,22	137,89	139,11	137,00	140,56	140,11	140,67	1,51	1,08
15,0	154,16	153,91	153,03	155,13	153,59	155,92	153,37	1,13	0,73
20,0	158,91	159,34	156,79	161,35	157,69	160,25	158,02	1,71	1,08
30,0	161,47	161,58	158,90	163,81	161,03	163,27	160,25	1,85	1,14
40,0	161,57	161,93	159,68	164,40	160,82	162,41	160,15	1,73	1,07
50,0	162,25	162,73	160,47	164,32	160,72	163,98	161,27	1,67	1,03
60,0	161,96	161,63	159,36	164,90	160,84	160,78	164,28	2,17	1,34
0,0									
0,0									
0,0									
0,0									
0,0									



M 10.07.97

066

19.06.97

Dissolution comprimés LF178TER

lot C0200 - 160mg

LSMa GOSH - 120TPM

1 - Préparation du milieu de dissolution.

cf page 062

2 - Pesée des lots de dissolution - balance GALOG5

1 litre de LSmG GOSH pèse 1002,0g

19.06.97

17:01:54

Code

1

Code

3

Code

5

0.00 g

0.00 g

0.00 g

698.8 g Tare

767.0 g Tare

760.5 g Tare

0.00 g

0.00 g

0.00 g

1002.0 g Net

1002.0 g Net

1002.0 g Net

0.00 g

0.00 g

0.00 g

1700.8 g Bunk

1769.0 g Bunk

1762.5 g Bunk

Code

2

Code

4

Code

6

0.00 g

0.00 g

0.00 g

768.8 g Tare

753.0 g Tare

760.8 g Tare

~~0.01 g~~ Pe

0.00 g

0.00 g

0.00 g

1002.0 g Net

1002.0 g Net

1002.0 g Net

0.00 g

0.00 g

0.00 g

1755.0 g Bunk  
PC1762.8 g Bunk  
PC1770.8 g Bunk  
PC

FOURNIER 1001769

Highly Confidential  
Subject to  
Protective Order3 - Conditions de dissolution.

Dissolvest Solax AT7. GAL091.

Température 37°C ± 0,5°C PC 20.06.97 sc 20/06/97

Agitation : 120TPM PC 20.06.97 sc 20/06/97

M10.07.97

# 4- Pesée des comprimés - Balance GAL 205.

067

20.06.97 LF178TER  
Code 09:08:13  
C0200

0.0 mg  
705.7 mg  
0.0 mg  
719.7 mg  
0.0 mg  
709.2 mg  
0.0 mg  
705.5 mg  
0.0 mg  
705.2 mg  
0.0 mg  
710.6 mg

RC

## 5- Mesures -

Sur spectrophotomètre UVIKON 922 - GAL 233 - en cuve de 2 mm de trajet optique à 290 nm.

290NM 2MM LF178TER LOT C0200 0.05M 120 TPM

06-20-1997 10:58

Lambda No. Valeur\_E

290.0	1	0.0001_1	Au/Au
290.0	2	-0.0023_1	LSNa/LNa
290.0	3	-0.0037_1	
290.0	4	-0.0000_1	
290.0	5	-0.0029_1	0 min
290.0	6	-0.0041_1	
290.0	7	-0.0013_1	
290.0	8	-0.0041_1	
290.0	9	1.2375_1	
290.0	10	1.2499_1	
290.0	11	1.2729_1	10 min
290.0	12	1.2681_1	
290.0	13	1.2715_1	
290.0	14	1.2872_1	
290.0	15	1.3836_1	
290.0	16	1.3826_1	
290.0	17	1.3962_1	15 min
290.0	18	1.3887_1	
290.0	19	1.3816_1	
290.0	20	1.4098_1	
290.0	21	1.4177_1	
290.0	22	1.4226_1	
290.0	23	1.4362_1	20 min
290.0	24	1.4279_1	
290.0	25	1.4165_1	
290.0	26	1.4441_1	
290.0	27	1.4412_1	
290.0	28	1.4464_1	
290.0	29	1.4548_1	30 min
290.0	30	1.4357_1	
290.0	31	1.4167_1	
290.0	32	1.4539_1	

290.0	33	1.4205_1	
290.0	34	1.4389_1	
290.0	35	1.4445_1	40 min
290.0	36	1.4282_1	
290.0	37	1.4236_1	
290.0	38	1.4489_1	
290.0	39	1.4258_1	
290.0	40	1.4295_1	
290.0	41	1.4319_1	50 min
290.0	42	1.4237_1	
290.0	43	1.4169_1	
290.0	44	1.4460_1	
290.0	45	1.4180_1	
290.0	46	1.4230_1	
290.0	47	1.4299_1	60 min
290.0	48	1.4233_1	
290.0	49	1.4097_1	
290.0	50	1.4377_1	

RC

UVIKON 922

RC Opérateur

FOURNIER 1001770

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
 DATE : 20/06/97  
 APPAREIL : GAL091GAL233  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2 mm

TITRE : LF178TER comprimés lot C0200 - 0,05M 120tpm  
 N° CAHIER : LF178ter dissolution n°2 page 066  
 FICHER : m:\commun\glnq\lf178ter\dissolution\lot C0200 0,05M 120tpm  
 ELUANT : LNa 0,05M  
 AGITATION : 120 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001771

Highly Confidential  
 Subject to  
 Protective Order

Témoins 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

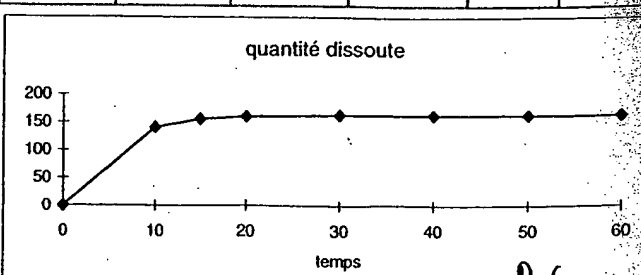
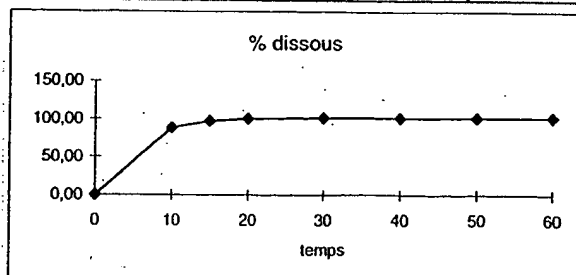
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,238	1,25	1,273	1,268	1,272	1,287
15	1000	1,384	1,383	1,396	1,389	1,382	1,41
20	1000	1,418	1,423	1,436	1,428	1,417	1,444
30	1000	1,441	1,446	1,455	1,436	1,417	1,454
40	1000	1,421	1,439	1,445	1,428	1,424	1,449
50	1000	1,426	1,43	1,432	1,424	1,417	1,446
60	1000	1,418	1,423	1,43	1,423	1,41	1,438

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	87,82	85,97	86,81	88,40	88,06	88,33	89,38	1,23	1,40
15,0	97,01	96,54	96,48	97,39	96,90	96,41	98,36	0,75	0,78
20,0	100,07	99,38	99,73	100,65	100,09	99,32	101,21	0,75	0,75
30,0	101,52	101,47	101,83	102,47	101,14	99,82	102,41	0,98	0,97
40,0	101,52	100,58	101,84	102,28	101,08	100,79	102,57	0,82	0,81
50,0	101,66	101,42	101,72	101,88	101,30	100,80	102,86	0,69	0,68
60,0	101,78	101,36	101,73	102,24	101,73	100,81	102,81	0,69	0,68
0,0									
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	140,52	137,56	138,89	141,44	140,89	141,33	143,00	1,96	1,40
15,0	155,22	154,47	154,36	155,82	155,04	154,26	157,38	1,21	0,78
20,0	160,10	159,01	159,57	161,04	160,14	158,92	161,94	1,19	0,75
30,0	162,44	162,36	162,92	163,95	161,83	159,71	163,86	1,57	0,97
40,0	162,44	160,93	162,95	163,64	161,73	161,27	164,11	1,31	0,81
50,0	162,66	162,28	162,75	163,00	162,08	161,28	164,58	1,11	0,68
60,0	162,85	162,18	162,76	163,58	162,76	161,29	164,49	1,11	0,68
0,0									
0,0									
0,0									
0,0									
0,0									



M. 10.07.97 B.C.

20.06.97

Dissolution LF 178TER comprimés

069

lot 234 - 100g

LSNa 0,025M - 75TPM

## 1. Préparation du milieu de dissolution. balance GAL 065

20.06.97 Eau purifiée 11:03:18

0.00 g

0.00 g

642.4 g Tare

645.2 g

0.00 g

0.00 g

2713.6 g Net

2630.4 g

0.00 g

0.00 g

3356.0 g Bunt

3275.6 g

0.00 g

644.8 g Tare

0.00 g

2466.3 g Net

20.06.97 11:09:37

0.00 g

3111.1 g Bunt

RC

Total de l'eau purifiée pesée

2713,6 g

+ 2466,3 g

+ 2630,4 g

7810,3 g

Pesée de LSNa milieu à 0,025 M  $P_{LSNa} = 288,4g$  $7810,3 \times 0,025 \times 288,4 = 56,31g$ 

1000

20.06.97 LSNa 11:22:52  
Code AAR 1547

0.00 g

91.90 g Tare

0.00 g

56.31 g Net

0.00 g

148.21 g Bunt  
RC

FOURNIER 1001772

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## 2. Pesée des Bols de dissolution - Balance GAL 065

1 litre de LSNa 0,025 M pèse 1001,0g.

20.06.97 13:01:34

1

0.00 g

698.8 g Tare

0.00 g

1001.0 g Net

0.00 g

1699.8 g Bunt

2

Code

0.00 g

768.8 g Tare

0.00 g

1001.0 g Net

0.00 g

1769.8 g Bunt

3

Code

0.00 g

760.5 g Tare

0.00 g

1001.0 g Net

0.00 g

1761.5 g Bunt

12/10/07.44

070

Code 4

0.00 g  
767.0 g Tare  
0.00 g  
1001.0 g Net  
0.00 g  
1768.0 g Bunt  
0.00 g

Code 5

0.00 g  
760.8 g Tare  
0.00 g  
1001.0 g Net  
0.00 g  
1761.8 g Bunt

Code 6

0.00 g  
753.0 g Tare  
0.00 g  
1001.0 g Net  
0.00 g  
1754.0 g Bunt

3- Conditions de dissolution -

Dissoluteur Sotax AT7 - GAL 091

Température  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  Re 20.06.97 2/6/97 ALAgitation  $30^{\text{Re}}$  75 TPM Re 20.06.97 2/6/97 AL4- Pesée des comprimés - Balance GAL 205

20.06.97 16/18/19/21/14:11:14

Code

Lot 234

0.0 mg

563.3 mg

~~0.2 mg~~ AL

0.0 mg

559.4 mg

0.0 mg

552.8 mg

0.0 mg

564.6 mg

0.0 mg

567.6 mg

0.0 mg

557.7 mg

5- Lectures -

Sur spectrophotomètre UVIKON 922 GAL 233 en cuve de  
2 mm de trajet optique à 290 nm.

FOURNIER 1001773  
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M 10.07.97

071

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER LOT 234 0.025M 75 TPM /

06-20-1997 16:39

Lambda	No.	Valeur_E
290.0	1	0.0004_1 <i>Pu/Au</i>
290.0	2	-0.0003_1 <i>is Na/K</i>
290.0	3	-0.0005_1
290.0	4	-0.0008_1
290.0	5	-0.0005_1 <i>10 min</i>
290.0	6	-0.0005_1
290.0	7	0.0005_1
290.0	8	-0.0004_1
290.0	9	0.5612_1
290.0	10	0.5935_1
290.0	11	0.6157_1 <i>10 min</i>
290.0	12	0.5616_1
290.0	13	0.5977_1
290.0	14	0.6050_1
290.0	15	0.6977_1
290.0	16	0.7068_1
290.0	17	0.7001_1 <i>15 min</i>
290.0	18	0.6865_1
290.0	19	0.7159_1
290.0	20	0.7015_1
290.0	21	0.7555_1
290.0	22	0.7505_1
290.0	23	0.7397_1 <i>20 min</i>
290.0	24	0.7408_1
290.0	25	0.7641_1
290.0	26	0.7437_1
290.0	27	0.8131_1
290.0	28	0.7999_1
290.0	29	0.7860_1 <i>20 min</i>
290.0	30	0.7981_1
290.0	31	0.8157_1
290.0	32	0.7944_1
290.0	33	0.8443_1
290.0	34	0.8307_1
290.0	35	0.8083_1 <i>40 min</i>
290.0	36	0.8304_1
290.0	37	0.8466_1
290.0	38	0.8220_1
290.0	39	0.8636_1
290.0	40	0.8452_1
290.0	41	0.8264_1 <i>50 min</i>
290.0	42	0.8489_1
290.0	43	0.8569_1
290.0	44	0.8416_1
290.0	45	0.8835_1
290.0	46	0.8549_1
290.0	47	0.8382_1 <i>60 min</i>
290.0	48	0.8648_1
290.0	49	0.8734_1
290.0	50	0.8519_1
290.0	51	0.9103_1
290.0	52	0.8905_1
290.0	53	0.8706_1 <i>120 min</i>
290.0	54	0.9031_1
290.0	55	0.9032_1
290.0	56	0.8849_1

FOURNIER 1001774

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M 10.07.47



# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
 DATE : 20/06/97  
 APPAREIL : GAL091GAL233  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2 mm

TITRE : LF178TER comprimés lot PK 234 - 0,025M 75tpm  
 N° CAHIER : U178ter dissolution n°2 page 069  
 FICHIER : m:\commun\glnq\U178ter\dissolution\lot PK 234 0,025M 75tpm  
 ELUANT : LNa 0,025M  
 AGITATION : 75 TPM

## PRÉPARATION DES ECHANTILLONS

masse théorique	100
dosage théorique	100 en mg

masse de la prise d'essai  
 quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
100,00	100,00	100,00	100,00	100,00	100,00
100,00	100,00	100,00	100,00	100,00	100,00

FOURNIER 1001775

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

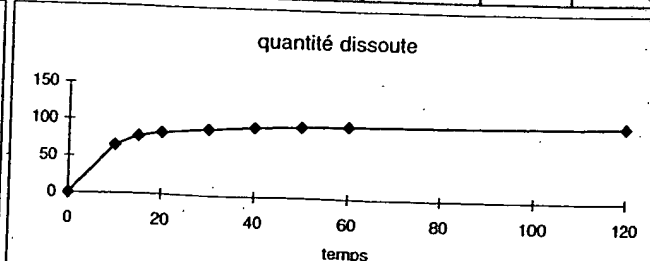
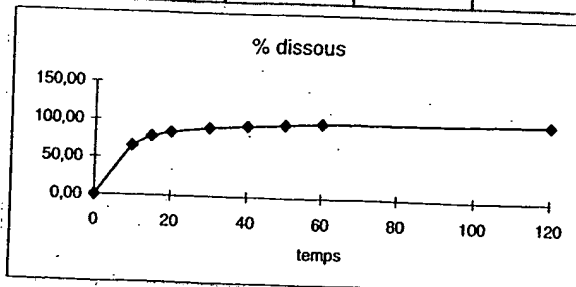
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,561	0,594	0,616	0,562	0,598	0,605
15	1000	0,698	0,707	0,7	0,687	0,716	0,702
20	1000	0,756	0,751	0,74	0,741	0,764	0,744
30	1000	0,813	0,8	0,786	0,798	0,816	0,794
40	1000	0,844	0,831	0,808	0,83	0,847	0,822
50	1000	0,864	0,845	0,826	0,849	0,857	0,842
60	1000	0,884	0,855	0,838	0,865	0,873	0,852
120	1000	0,91	0,891	0,871	0,903	0,903	0,885

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	65,48	62,33	66,00	68,44	62,44	66,44	67,22	2,54	3,87
15,0	78,29	77,87	78,89	78,12	76,65	79,89	78,34	1,08	1,38
20,0	83,98	84,70	84,17	82,95	83,03	85,62	83,39	1,05	1,25
30,0	90,15	91,45	90,03	88,48	89,77	91,82	89,36	1,27	1,41
40,0	93,84	95,35	93,92	91,36	93,77	95,72	92,91	1,60	1,71
50,0	96,17	98,04	95,94	93,81	96,34	97,30	95,59	1,47	1,53
60,0	98,20	100,74	97,52	95,60	98,59	99,55	97,17	1,83	1,86
120,0	102,30	104,12	101,99	99,73	103,30	103,37	101,31	1,62	1,58
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	65,48	62,33	66,00	68,44	62,44	66,44	67,22	2,54	3,87
15,0	78,29	77,87	78,89	78,12	76,65	79,89	78,34	1,08	1,38
20,0	83,98	84,70	84,17	82,95	83,03	85,62	83,39	1,05	1,25
30,0	90,15	91,45	90,03	88,48	89,77	91,82	89,36	1,27	1,41
40,0	93,84	95,35	93,92	91,36	93,77	95,72	92,91	1,60	1,71
50,0	96,17	98,04	95,94	93,81	96,34	97,30	95,59	1,47	1,53
60,0	98,20	100,74	97,52	95,60	98,59	99,55	97,17	1,83	1,86
120,0	102,30	104,12	101,99	99,73	103,30	103,37	101,31	1,62	1,58
0,0									
0,0									
0,0									
0,0									



m 10.07.97 AC

24.06.97

Dissolution LF178TER

073

lot 158 - 100mg X2 = 200 mg

LSNa 0,025M - 75.TPM

# 1 - Préparation du milieu de dissolution. Balance GAL065

24.06.97 Eau 12:57:42

0.00 g purifiée

642.4 g Tare

0.00 g

2424.7 g Net

0.00 g

3067.0 g Bulk

0.00 g

644.4 g Tare

0.00 g

2607.8 g Net

0.00 g

3252.2 g Bulk

~~0.01 g RC~~

0.00 g

644.6 g Tare

0.00 g

2428.0 g Net

0.00 g

3072.5 g Bulk RC

Tota Pde l'eau purifiée pesée:

$$2424,7 + 2607,8 + 2428,0 = 7460,5g$$

Pesée de LSNa pour un milieu à  
0,025 M - PM<sub>LSNa</sub> = 288,4g

$$\frac{7460,5}{1000} \times 0,025 \times 288,4 = 53,79g$$

24.06.97 LSNa 13:11:02

Code ARR1547,

0.00 g

161.47 g Tare

0.00 g

53.79 g Net

0.00 g

215.26 g Bulk

RC

## 2 - Pesée des lots de dissolution - Balance GAL065

1 l de LSNa 0,025M pèse 1001,0 g -

FOURNIER 1001776

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M. 10.07.97

074

24.06.97

13:23:53

Code

1

Code

4

Code

5

0.00 g

760.8 g Tare

0.00 g

1001.0 g Net

0.00 g

1761.8 g Buik  
RC 2

Code

0.00 g

760.5 g Tare

0.00 g

1001.0 g Net

0.00 g

1761.4 g Buik

0.00 g

698.8 g Tare

0.00 g

1001.0 g Net

0.00 g

1699.8 g Buik  
RC 3

Code

0.00 g

768.8 g Tare

0.00 g

1001.0 g Net

0.00 g

1769.8 g Buik

0.00 g

767.0 g Tare

0.00 g

1001.0 g Net

0.00 g

1768.0 g Buik

Code

6

0.00 g

753.0 g Tare

0.00 g

1001.0 g Net

0.00 g

1754.0 g Buik  
RC3 - Conditions de dissolution.

Dissolutesolax ATF - GAL091.

Température  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  sz 24/06/97 RC 24.06.97

Agitation 75 TPM - sz 24/06/97 RC 24.06.97

4 - Pesée des comprimés - Balance GAL 205 -24.06.97 16:17:26  
Code Lot 158

0.0 mg

1128.2 mg

0.0 mg

1123.5 mg

0.0 mg

1116.2 mg

0.0 mg

1103.1 mg

0.0 mg

1120.5 mg

0.0 mg

1125.3 mg

RC

5 - Lectures -Sur spectrophomètre UV/KON 922 - GAL233 en cuve  
de 2mm de trajet optique à 290 nm.FOURNIER 1001777  
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M 10.07.97

06-24-1997 17:38

Lambda	No.	Valeur_E	
290.0	1	0.0001_1	Ain/Ai
290.0	2	-0.0007_1	LSNa/LSNa
290.0	3	-0.0005_1	
290.0	4	0.0019_1	
290.0	5	-0.0007_1	0min
290.0	6	0.0000_1	
290.0	7	-0.0007_1	
290.0	8	-0.0010_1	
290.0	9	1.2177_1	
290.0	10	1.1791_1	
290.0	11	1.2255_1	10min
290.0	12	1.1895_1	
290.0	13	1.2229_1	
290.0	14	1.2462_1	
290.0	15	1.4427_1	
290.0	16	1.3853_1	
290.0	17	1.4347_1	15min
290.0	18	1.3762_1	
290.0	19	1.4121_1	
290.0	20	1.4372_1	
290.0	21	1.5317_1	
290.0	22	1.4886_1	
290.0	23	1.5198_1	20min
290.0	24	1.4686_1	
290.0	25	1.5076_1	
290.0	26	1.5284_1	
290.0	27	1.6221_1	
290.0	28	1.5898_1	
290.0	29	1.6101_1	30min
290.0	30	1.5709_1	
290.0	31	1.5957_1	
290.0	32	1.6137_1	
290.0	33	1.6733_1	
290.0	34	1.6412_1	
290.0	35	1.6555_1	40min
290.0	36	1.6256_1	
290.0	37	1.6518_1	
290.0	38	1.6567_1	
290.0	39	1.7056_1	
290.0	40	1.6860_1	
290.0	41	1.6889_1	50min
290.0	42	1.6465_1	
290.0	43	1.6830_1	
290.0	44	1.6967_1	
290.0	45	1.7406_1	
290.0	46	1.7022_1	
290.0	47	1.6889_1	60min
290.0	48	1.6854_1	
290.0	49	1.7048_1	
290.0	50	1.7152_1	
290.0	51	1.8002_1	
290.0	52	1.7837_1	
290.0	53	1.7878_1	120min
290.0	54	1.7584_1	
290.0	55	1.7794_1	
290.0	56	1.7902_1	

AC

FOURNIER 1001778

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M. 10.07.97

## DISSOLUTION

m:\commun\glng\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
 DATE : 24/06/97  
 APPAREIL : GAL091GAL233  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2 mm

TITRE : LF178TER comprimés lot PK 158 - 0,025M 75tpm 2 comprimés  
 N° CAHIER : LF178ter dissolution n°2 page 073  
 FICHER : m:\commun\glng\lf178ter\dissolution\lot PK 158 0,025M 75tpm 2 comprimés  
 ELUANT : LSNa 0,025M  
 AGITATION : 75 tpm

## PREPARATION DES ECHANTILLONS

masse théorique 200  
 dosage théorique 200 en mg

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
200,00	200,00	200,00	200,00	200,00	200,00
200,00	200,00	200,00	200,00	200,00	200,00

masse de la prise d'essai  
 quantité de principe actif

FOURNIER 1001779

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

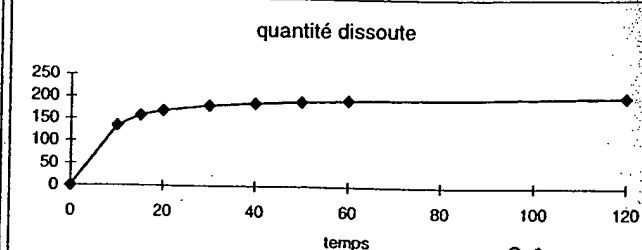
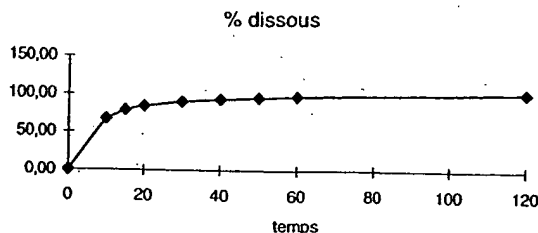
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,218	1,179	1,226	1,19	1,223	1,246
15	1000	1,443	1,385	1,435	1,376	1,412	1,437
20	1000	1,532	1,489	1,52	1,469	1,508	1,528
30	1000	1,622	1,59	1,61	1,571	1,596	1,614
40	1000	1,673	1,641	1,656	1,626	1,652	1,657
50	1000	1,706	1,686	1,689	1,647	1,683	1,697
60	1000	1,741	1,702	1,689	1,685	1,705	1,715
120	1000	1,8	1,784	1,788	1,758	1,779	1,79

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	67,43	67,67	65,50	68,11	66,11	67,94	69,22	1,38	2,04
15,0	78,93	80,51	77,27	80,06	76,78	78,78	80,18	1,60	2,02
20,0	84,49	85,85	83,43	85,18	82,32	84,51	85,63	1,37	1,63
30,0	90,07	91,28	89,46	90,61	88,40	89,82	90,84	1,05	1,17
40,0	93,31	94,56	92,73	93,61	91,89	93,37	93,67	0,91	0,97
50,0	95,64	96,86	95,69	95,90	93,51	95,55	96,36	1,15	1,20
60,0	97,31	99,28	97,05	96,37	96,08	97,24	97,83	1,15	1,18
120,0	102,06	103,04	102,08	102,34	100,60	101,83	102,47	0,82	0,81
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	134,85	135,33	131,00	136,22	132,22	135,89	138,44	2,75	2,04
15,0	157,86	161,01	154,54	160,13	153,55	157,57	160,36	3,19	2,02
20,0	168,98	171,70	166,87	170,37	164,65	169,02	171,27	2,75	1,63
30,0	180,13	182,55	178,92	181,21	176,80	179,64	181,67	2,11	1,17
40,0	186,61	189,12	185,47	187,22	183,78	186,74	187,35	1,82	0,97
50,0	191,29	193,72	191,38	191,80	187,02	191,11	192,71	2,30	1,20
60,0	194,61	198,55	194,09	192,74	192,16	194,49	195,66	2,30	1,18
120,0	204,12	206,08	204,15	204,68	201,20	203,66	204,94	1,65	0,81
0,0									
0,0									
0,0									
0,0									



M-10.07.07 RC

24.06.97

Dissolution LESTER - comprimés

077

lot 2398 / 01 RG - 14 kg

LSNa 905M - 120TPM

## 1. Préparation du milieu dissolution - Balance GAL 065

24.06.97 Eau 16:48:56  
0.00 g purifiée

642.4 g Tare

0.00 g

2514.3 g Net

0.00 g

3156.7 g Buik

0.00 g

645.0 g Tare

~~0.01 g~~ RC

0.00 g

2691.7 g Net

0.00 g

3336.8 g Buik

0.00 g

645.0 g Tare

~~0.01 g~~ RC

0.00 g

2487.8 g Net

0.00 g

3132.8 g Buik  
RC

0.00 g

644.9 g Tare

0.00 g

2446.1 g Net

0.00 g

3090.9 g Buik

0.00 g

645.2 g Tare

0.00 g

2858.7 g Net

0.00 g

3503.8 g Buik  
RCTotal de l'eau purifiée  
pesée -

2514.3 g

+ 2691.7 g

+ 2487.8 g

+ 2446.1 g

+ 2858.7 g

12998.6 g ✓

Pesée de LSNa pour un milieu à 905M

PM LSNa = 288.4 g

$$\frac{12998.6}{1000} \times 905 \times 288.4 = 187.44 \text{ g}$$
24.06.97 LSNa 17:19:41  
Code APR 1547

0.00 g

143.22 g Tare

0.00 g

187.45 g Net

0.00 g

330.67 g Buik

RC

## 2. Pesée des bds de dissolution - Balance GAL 065

un litre de LSNa 905 M pèse 1008.0 g -

M 10.07.97

FOURNIER 1001780

Highly Confidential  
Subject to  
Protective Order

078

25.06.97  
Code09:12:07  
1

Code

3

Code

5

0.00 g

0.00 g

0.00 g

760.8 g Tare

767.0 g Tare

760.5 g Tare

0.00 g

0.00 g

0.00 g

1002.0 g Net

1002.0 g Net

1002.0 g Net

0.00 g

0.00 g

0.00 g

1762.8 g Bulk

1769.0 g Bulk

1762.4 g Bulk

Code

2

Code

4

Code

6

0.00 g

0.00 g

0.00 g

698.8 g Tare

753.0 g Tare

760.8 g Tare

0.00 g

0.00 g

0.00 g

1002.0 g Net

1002.0 g Net

1002.0 g Net

0.00 g

0.00 g

0.00 g

RC 1700.8 g Bulk

RC 1755.0 g Bulk

RC 1770.8 g Bulk

3. Conditions de dissolution

Dissolvent AT7 - GAL 091

Température  $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$   $\approx 25/06/97$  RC 25.06.97Agitation 120 TPM  $\approx 25/06/97$  RC 25.06.974. Pesée des comprimés - Balance GAL 205

LEISTER

25.06.97 10:58:54

Code RG-2398/01-146g

0.0 mg

695.0 mg

0.0 mg

691.7 mg

0.0 mg

691.2 mg

0.0 mg

700.2 mg

0.0 mg

697.8 mg

0.0 mg

697.3 mg

FOURNIER 1001781

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Protective Order

M 10.07.97

# 5 - Lectures

079

Sur spectrophotomètre UVIKON 922 GAL 233 dans des cuves de 2 mm de trajet optique à 290 nm.

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER 2398/01RG 14KG LSNA0.05M 120TPM /

06-25-1997 12:11

Lambda	No.	Valeur_E
290.0	1	0.0000_1 <i>A<sub>in</sub> / A<sub>in</sub></i>
290.0	2	-0.0006_1 <i>LS<sub>no</sub> / LS<sub>no</sub></i>
290.0	3	-0.0002_1
290.0	4	-0.0010_1
290.0	5	-0.0009_1 <i>0 min</i>
290.0	6	-0.0012_1
290.0	7	-0.0012_1
290.0	8	-0.0012_1
290.0	9	1.2868_1
290.0	10	1.2887_1
290.0	11	1.2892_1 <i>10 min</i>
290.0	12	1.3017_1
290.0	13	1.3034_1
290.0	14	1.3104_1
290.0	15	1.4249_1
290.0	16	1.4072_1
290.0	17	1.3864_1 <i>15 min</i>
290.0	18	1.4285_1
290.0	19	1.4013_1
290.0	20	1.4080_1
290.0	21	1.4602_1
290.0	22	1.4419_1
290.0	23	1.4042_1 <i>20 min</i>
290.0	24	1.4556_1
290.0	25	1.4258_1
290.0	26	1.4338_1
290.0	27	1.4821_1
290.0	28	1.4618_1
290.0	29	1.4175_1 <i>30 min</i>
290.0	30	1.4739_1
290.0	31	1.4341_1
290.0	32	1.4444_1
290.0	33	1.4613_1
290.0	34	1.4486_1
290.0	35	1.4131_1 <i>40 min</i>
290.0	36	1.4700_1
290.0	37	1.4356_1
290.0	38	1.4334_1
290.0	39	1.4714_1
290.0	40	1.4402_1
290.0	41	1.4134_1 <i>60 min</i>
290.0	42	1.4585_1
290.0	43	1.4249_1
290.0	44	1.4415_1

KONTRON INSTRUMENTS UVIKON 922

Opérateur .....

PC

M 10.07.07

FOURNIER 1001782

Highly Confidential  
Subject to  
Protective Order



080

**DISSOLUTION**

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

**CONDITIONS ET MODES OPERATOIRES**

OPERATEUR : ROSSELIN C  
 DATE : 25/06/97 /  
 APPAREIL : GAL091GAL233 /  
 LONGUEUR D'ONDE : 290 nm /  
 CUVE en mm : 2 mm /

TITRE : LF178TER comprimés lot 2398/01 RG 14 Kg - 0,05M 120tpm  
 N° CAHIER : U178ter dissolution n°2 page 077 /  
 FICHIER : m:\commun\glnq\lf178ter\dissolution\ot 2398RG01 14 Kg 0,05M 120tpm  
 ELUANT : LSNa 0,05M /  
 AGITATION : 120 tpm /

**PREPARATION DES ECHANTILLONS**

masse théorique 160 /  
 dosage théorique 160 en mg

masse de la prise d'essai  
 quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

**FOURNIER 1001783**

Highly Confidential  
 Subject to  
 Protective Order

Témoin 100mg/l 0,900

**SUIVI DE LA DISSOLUTION**

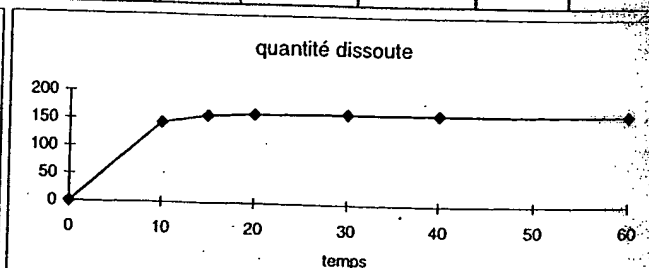
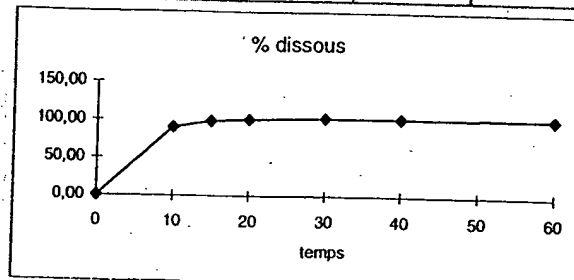
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,287 /	1,289 /	1,289 /	1,302 /	1,303 /	1,31 /
15	1000	1,425 /	1,407 /	1,386 /	1,429 /	1,401 /	1,408 /
20	1000	1,46 /	1,442 /	1,404 /	1,456 /	1,426 /	1,434 /
30	1000	1,482 /	1,462 /	1,418 /	1,474 /	1,434 /	1,444 /
40	1000	1,461 /	1,449 /	1,413 /	1,47 /	1,436 /	1,433 /
60	1000	1,471 /	1,44 /	1,413 /	1,459 /	1,425 /	1,442 /

**RESULTATS EN % DISSOUS**

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	90,05	89,38	89,51	89,51	90,42	90,49	90,97	0,66	0,74
15,0	98,32	99,41	98,16	96,70	99,69	97,74	98,23	1,10	1,12
20,0	100,73	102,33	101,08	98,43	102,06	99,97	100,53	1,44	1,43
30,0	102,30	104,37	102,96	99,89	103,81	101,02	101,72	1,72	1,68
40,0	102,20	103,42	102,57	100,03	104,05	101,65	101,46	1,46	1,42
60,0	102,56	104,62	102,45	100,52	103,80	101,39	102,58	1,50	1,47
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									

**RESULTATS EN QUANTITE DISSOUE**

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	144,07	143,00	143,22	143,22	144,67	144,78	145,56	1,06	0,74
15,0	157,31	159,05	157,05	154,72	159,50	156,39	157,17	1,76	1,12
20,0	161,17	163,73	161,72	157,49	163,30	159,95	160,84	2,30	1,43
30,0	163,67	166,98	164,74	159,82	166,10	161,63	162,75	2,75	1,68
40,0	163,52	165,47	164,11	160,05	166,48	162,65	162,33	2,33	1,42
60,0	164,10	167,40	163,92	160,84	166,07	162,22	164,13	2,41	1,47
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									



m 10.07.97

25.06.97

Dissolution LF 178TER comprimés  
lot 2401/01 PG - 20kg  
LSNa 0,05M - 120TPM

081

1. Préparation du milieu  
 cf page 077.

2. Pesée des lots de dissolution Balance GFL 065.  
 1 litre de LSNa 0,05M pèse 1002,0g.

25.06.97 13:18:33  
 Code 1

0.00 g  
 698.8 g Tare  
 0.00 g  
 1002.0 g Net  
~~0.01 g~~ be  
 0.00 g  
 1700.8 g Bulk

Code 2

0.00 g  
 768.8 g Tare  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1770.8 g Bulk  
 RC

Code 3

0.00 g  
 753.0 g Tare  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1755.0 g Bulk

Code 4

0.00 g  
 767.0 g Tare  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1769.0 g Bulk  
 RC

Code 5

0.00 g  
 760.8 g Tare  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1762.8 g Bulk

Code 6

0.00 g  
 760.5 g Tare  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1762.4 g Bulk  
 RC

3. Conditions de dissolution

Dissolutes AT7 - GAL 091 -

Température:  $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  RC 25.06.97 ~~25/06/97~~

Agitation: 120TPM RC 25.06.97 ~~25/06/97~~

M 10.07.97

FOURNIER 1001784  
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#### 4. Pesée des comprimés - Balance GAL205

LF178TER  
25.05.97 15:00:14  
Code RG 2401-01-20Kg

0.0 mg  
698.3 mg  
0.0 mg  
697.2 mg  
0.0 mg  
689.8 mg  
0.0 mg  
700.6 mg  
0.0 mg  
695.2 mg  
0.0 mg  
697.9 mg

AL

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER 2401/01RG 20KG LSNA0.05M 120TPM

#### 5. Lectures

06-25-1997 16:34

Sur spectrophotomètre  
UVIKON 922 GAL 233 en  
cuve de 2mm de trajet  
optique à 290 nm -

Lambda	No.	Valeur_E
290.0	1	0.0003_1
290.0	2	-0.0010_1
290.0	3	-0.0007_1
290.0	4	-0.0009_1
290.0	5	-0.0009_1
290.0	6	-0.0010_1
290.0	7	-0.0005_1
290.0	8	-0.0009_1
290.0	9	0.9383_1
290.0	10	1.0131_1
290.0	11	1.0153_1
290.0	12	1.0253_1
290.0	13	1.0739_1
290.0	14	1.0835_1
290.0	15	1.3008_1
290.0	16	1.3130_1
290.0	17	1.3146_1
290.0	18	1.3349_1
290.0	19	1.3288_1
290.0	20	1.3395_1
290.0	21	1.4168_1
290.0	22	1.3997_1
290.0	23	1.3989_1
290.0	24	1.4157_1
290.0	25	1.3956_1
290.0	26	1.4045_1
290.0	27	1.4722_1
290.0	28	1.4508_1
290.0	29	1.4448_1
290.0	30	1.4639_1
290.0	31	1.4328_1
290.0	32	1.4440_1
290.0	33	1.4739_1
290.0	34	1.4549_1
290.0	35	1.4474_1
290.0	36	1.4710_1
290.0	37	1.4475_1
290.0	38	1.4586_1
290.0	39	1.4797_1
290.0	40	1.4627_1
290.0	41	1.4624_1
290.0	42	1.4834_1
290.0	43	1.4598_1
290.0	44	1.4534_1

Air / Air  
LSNa / LSNa

0mm

10mm

15mm

20mm

30mm

40mm

60mm

FOURNIER 1001785

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

AL

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
 DATE : 25/06/97  
 APPAREIL : GAL091GAL233  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2 mm

TITRE : LF178TER comprimés lot 2401/01 RG 20 Kg - 0,05M 120tpm  
 N° CAHIER : LF178ter dissolution n°2 page 081  
 FICHER : m:\commun\glnq\lf178ter\dissolution\lot 2401RG01 20 Kg 0,05M 120tpm  
 ELUANT : LNa 0,05M  
 AGITATION : 120 tpm

## PREPARATION DES ECHANTILLONS

masse théorique 160 /  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001786

Highly Confidential  
 Subject to  
 Protective Order

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

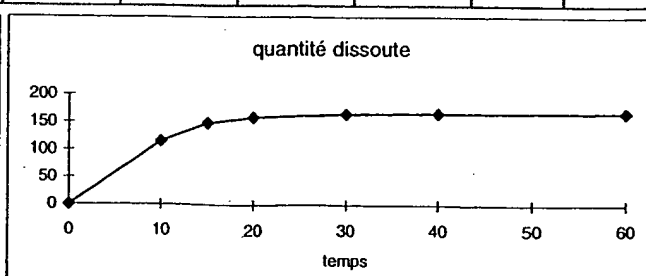
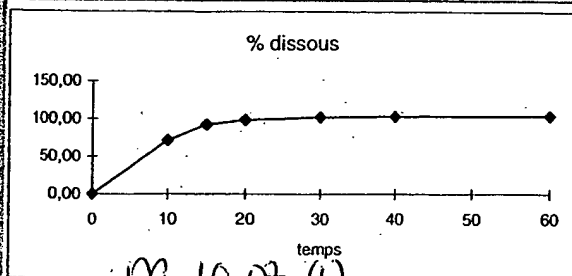
volume prélevé en ml 5							
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,938 /	1,013 /	1,015 /	1,025 /	1,074 /	1,084 /
15	1000	1,301 /	1,313 /	1,315 /	1,335 /	1,329 /	1,34 /
20	1000	1,417 /	1,4 /	1,399 /	1,416 /	1,396 /	1,405 /
30	1000	1,472 /	1,451 /	1,445 /	1,464 /	1,433 /	1,444 /
40	1000	1,474 /	1,455 /	1,447 /	1,471 /	1,448 /	1,459 /
60	1000	1,48 /	1,463 /	1,462 /	1,483 /	1,46 /	1,453 /

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	71,17	65,14	70,35	70,49	71,18	74,58	75,28	3,63	5,11
15,0	92,17	90,67	91,53	91,67	93,06	92,66	93,43	1,05	1,14
20,0	98,42	99,18	98,03	97,96	99,15	97,78	98,41	0,61	0,62
30,0	102,10	103,49	102,06	101,64	102,98	100,83	101,61	0,98	0,96
40,0	103,13	104,14	102,84	102,28	103,97	102,37	103,15	0,79	0,76
60,0	104,18	105,07	103,90	103,83	105,32	103,71	103,24	0,82	0,79
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	113,87	104,22	112,56	112,78	113,89	119,33	120,44	5,82	5,11
15,0	147,48	145,08	146,45	146,68	148,90	148,26	149,49	1,68	1,14
20,0	157,47	158,69	156,85	156,74	158,64	156,45	157,46	0,98	0,62
30,0	163,36	165,59	163,29	162,63	164,76	161,33	162,57	1,56	0,96
40,0	165,00	166,63	164,54	163,65	166,36	163,80	165,04	1,26	0,76
60,0	166,68	168,11	166,24	166,12	168,51	165,93	165,18	1,32	0,79
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									



27.06.97

Dissolution LF 178TER comprimés  
lot 2401/01 RG-15Kg  
LSNa 0,05M - 120 TPM

1. Préparation du milieu de dissolution - Balance GAL 065

27.06.97 Eau 08:52:57  
0.00 g purifiée

642.4 g ~~tare~~

0.00 g

2518.5 g Net

0.00 g

3160.9 g Brut

0.00 g

644.9 g Tare

0.00 g

2477.7 g Net

0.00 g

3122.5 g Brut

0.00 g

644.7 g Tare

0.00 g

1950.4 g Brut Net

0.00 g

2595.1 g Brut  
PC

Total de l'eau purifiée pesée

$$2518,5 + 2477,7 + 1950,4 = 6946,6 \text{ g}$$

Pesée de LSNa pour avoir un milieu  
à 0,05M -  $PM_{LSNa} = 28,4 \text{ g}$

$$\frac{6946,6}{1000} \times 0,05 \times 28,4 = 100,17 \text{ g}$$

LSNa.

27.06.97 09:06:22  
Code AAR 1547

0.00 g

163.60 g

0.00 g

100.17 g /

0.00 g

263.78 g

PC

2. Contrôle Pesée des lots de dissolution - Balance GAL 065

1 litre de LSNa 0,05 M pèse 1002,0 g -

FOURNIER 1001787  
Highly Confidential  
Subject to  
Protective Order

M 10.07.07

21.06.97  
Code

07:18:33  
1

Code

3

Code

5

085

0.00 g  
768.8 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1770.8 g Bunk

0.00 g  
760.5 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1762.5 g Bunk

0.00 g  
698.8 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1700.8 g Bunk

Code

2

Code

4

Code

6

0.00 g  
760.8 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1762.8 g Bunk  
RC

0.00 g  
767.0 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1769.0 g Bunk  
RC

0.00 g  
753.0 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1755.0 g Bunk  
RC

### 3. Conditions de dissolution

Dissoltest ATF Solvax GAL 091

Température 37.2 ± 0.5°C - RE 27.06.97 SE 27/06/97

Agitation 120TPM - RC 27.06.97 SE 27/06/97

LE17878

27.06.97 10:26:45  
Code RG 2401/01-15%

0.0 mg

695.9 mg

0.0 mg

689.4 mg

0.0 mg

695.3 mg

0.0 mg

694.1 mg

0.0 mg

694.7 mg

0.0 mg

695.8 mg

RC

M 10.07.97

FOURNIER 1001788  
Highly Confidential  
Subject to  
Protective Order

086

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER LOT 2401/01RG 15KG LSNA0.05M 120TPM

06-27-1997 11:43

Lambda	No.	Valeur_E
290.0	1	0.0004_1
290.0	2	0.0016_1
290.0	3	-0.0001_1
290.0	4	-0.0005_1
290.0	5	-0.0007_1
290.0	6	0.0000_1
290.0	7	-0.0007_1
290.0	8	-0.0007_1
290.0	9	1.2129_1
290.0	10	1.2293_1
290.0	11	1.2299_1
290.0	12	1.2424_1
290.0	13	1.2421_1
290.0	14	1.2420_1
290.0	15	1.3729_1
290.0	16	1.3716_1
290.0	17	1.3456_1
290.0	18	1.3654_1
290.0	19	1.3715_1
290.0	20	1.3837_1
290.0	21	1.3779_1
290.0	22	1.4146_1
290.0	23	1.3908_1
290.0	24	1.4007_1
290.0	25	1.4092_1
290.0	26	1.4218_1
290.0	27	1.4524_1
290.0	28	1.4433_1
290.0	29	1.4190_1
290.0	30	1.4235_1
290.0	31	1.4355_1
290.0	32	1.4481_1
290.0	33	1.4478_1
290.0	34	1.4329_1
290.0	35	1.4132_1
290.0	36	1.4258_1
290.0	37	1.4386_1
290.0	38	1.4458_1
290.0	39	1.4585_1
290.0	40	1.4443_1
290.0	41	1.4214_1
290.0	42	1.4402_1
290.0	43	1.4423_1
290.0	44	1.4395_1

LSNa/LSNa  
an / an

0mm

10mm

15mm

20mm

30mm

40mm

60mm

KONTRON INSTRUMENTS UVIKON 922

Opérateur

AL

FOURNIER 1001789

Highly Confidential  
Subject to  
Protective Order

m 10.07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

087

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 02/28/06/97  
APPAREIL : GAL091GAL233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2 mm

TITRE : LF178TER comprimés lot 2401/01 RG 15Kg - 0,05M 120 tpm  
N° CAHIER : LF178ter dissolution n°2 page 084  
FICHIER : m:\commun\glnq\lf178ter\dissolution\lot 2401rg01 15 kg 0,05M 120tpm  
ELUANT : LNa 0,05M  
AGITATION : 120 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/ 0,900

FOURNIER 1001790

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## SUIVI DE LA DISSOLUTION

TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,213	1,229	1,23	1,242	1,242	1,242
15	1000	1,373	1,372	1,346	1,365	1,372	1,384
20	1000	1,378	1,415	1,391	1,401	1,409	1,422
30	1000	1,452	1,443	1,419	1,424	1,436	1,448
40	1000	1,448	1,433	1,413	1,426	1,439	1,446
60	1000	1,459	1,444	1,421	1,44	1,442	1,44

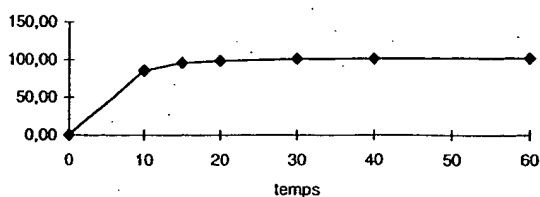
## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	85,63	84,24	85,35	85,42	86,25	86,25	86,25	0,80	0,94
15,0	95,47	95,77	95,70	93,90	95,22	95,71	96,54	0,88	0,92
20,0	98,31	96,59	99,17	97,49	98,20	98,75	99,66	1,13	1,15
30,0	101,18	102,21	101,60	99,92	100,28	101,12	101,96	0,92	0,91
40,0	101,48	102,44	101,41	100,00	100,91	101,83	102,33	0,92	0,91
60,0	102,46	103,70	102,67	101,04	102,38	102,53	102,41	0,85	0,83
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									

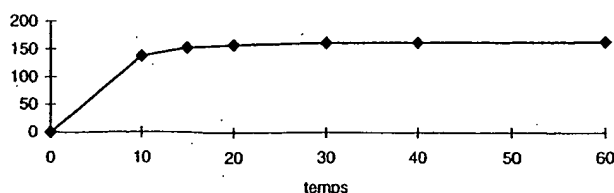
## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	137,00	134,78	136,56	136,67	138,00	138,00	138,00	1,28	0,94
15,0	152,76	153,23	153,13	150,24	152,36	153,13	154,47	1,41	0,92
20,0	157,30	154,55	158,67	155,99	157,12	158,01	159,46	1,81	1,15
30,0	161,89	163,54	162,56	159,87	160,45	161,79	163,14	1,48	0,91
40,0	162,37	163,90	162,26	159,99	161,46	162,92	163,72	1,48	0,91
60,0	163,93	165,92	164,27	161,67	163,81	164,05	163,86	1,36	0,83
0,0									
0,0									
0,0									
0,0									
0,0									
0,0									

% dissous



quantité dissoute



ML 10.07.97



1188

Dissolution LF178TER comprimés  
lot 2398/01 RG 14 Kg  
LSNa 0,1 M - 90TPH

1 - Préparation du milieu de dissolution - Balance GAL065

Eau  
 purifiée  
 30.06.97 15:14:24  
 0.00 g

642.5 g Tare

0.00 g

2729.5 g Net

0.00 g

3371.9 g Brut

0.00 g

645.2 g Tare

0.00 g

2674.6 g Net

0.00 g

3319.8 g Brut

0.00 g

644.9 g Tare

0.00 g

2664.4 g Net

0.00 g

3309.2 g Brut

15:14:24

0.00 g

644.7 g Tare

0.00 g

2678.7 g Net

0.00 g

3323.4 g Brut

0.00 g

645.0 g Tare

0.00 g

2899.3 g Net

0.00 g

3544.2 g Brut

RC

Pesée de LSNa pour un  
 milieu à 0,1 M - PH<sub>con</sub> = 288,4g

$13646,5 \times 0,1 \times 288,4 = 393,57g$   
 1000

RC

Total de l'eau purifiée pesée

2729,5 g

2674,6 g

2664,4 g

2678,7 g

2899,3 g

13646,5 g

LSNa

30.06.97

16:39:53

Code

ARR

1547

0.00 g

143.23 g

0.00 g

393.58 g

0.00 g

536.80 g

RC

2 - Pesée des bords de dissolution - Balance GAL065

1 litre de LSNa 0,1 M pèse 1004,0g

FOURNIER 1001791

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m 10.07.97

30.06.97  
Code17:07:03  
1

Code

3

Code

5

0.00 g

0.00 g

0.00 g

698.8 g Tare

768.8 g Tare

753.0 g Tare

0.00 g

0.00 g

0.00 g

1004.0 g Net,

1004.0 g Net,

1004.0 g Net,

0.00 g

0.00 g

0.00 g

1702.8 g Bunk

1772.8 g Bunk

1757.1 g Bunk

Code

2

Code

4

Code

6

0.00 g

0.00 g

0.00 g

760.8 g Tare

760.5 g Tare

767.0 g Tare

0.00 g

0.00 g

0.00 g

1004.0 g Net,

1004.0 g Net,

1004.0 g Net,

0.00 g

0.00 g

0.00 g

1764.9 g Bunk  
RC1764.5 g Bunk  
RC1771.0 g Bunk  
RC3. Conditions de dissolution

Dissolvest Solar AT7, CALOS1.

Température :  $87^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  RC 01.07.97 201/07/82

Agitation : 90TPM RC 01.07.97 201/07/82,

4. Pesée des comprimés - Balance CAL 205.

LF 178TER

01.07.97 10:34:31

Code RC 2398/01-14kg

0.0 mg

693.0 mg

0.0 mg

694.2 mg

0.0 mg

698.8 mg

0.0 mg

692.4 mg

0.0 mg

692.9 mg

0.0 mg

690.0 mg

RC

FOURNIER 1001792

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M 10.07.97

090

5. Lectures

Sur spectrophotométrie UVIKON 922 GHL233 en cuve de  
2 mm de trajet optique à 290 nm -

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER 2398/01RG 14KG LSNA 0.1M 90 TPM/

07-01-1997 11:38

Lambda	No.	Valeur_E
290.0	1	-0.0001_1 <i>Au/Air</i>
290.0	2	-0.0014_1 <i>LSNA / LSNA</i>
290.0	3	-0.0027_1
290.0	4	-0.0018_1
290.0	5	-0.0020_1 <i>0 min</i>
290.0	6	-0.0023_1
290.0	7	-0.0023_1
290.0	8	-0.0023_1
290.0	9	1.3510_1
290.0	10	1.3643_1
290.0	11	1.3582_1 <i>10 min</i>
290.0	12	1.3773_1
290.0	13	1.3665_1
290.0	14	1.3885_1
290.0	15	1.4558_1
290.0	16	1.4681_1
290.0	17	1.4686_1 <i>15 min</i>
290.0	18	1.4565_1
290.0	19	1.4326_1
290.0	20	1.4526_1
290.0	21	1.4715_1
290.0	22	1.4843_1
290.0	23	1.4790_1 <i>20 min</i>
290.0	24	1.4663_1
290.0	25	1.4434_1
290.0	26	1.4540_1
290.0	27	1.4677_1
290.0	28	1.4916_1
290.0	29	1.4772_1 <i>30 min</i>
290.0	30	1.4669_1
290.0	31	1.4459_1
290.0	32	1.4639_1

KONTRON INSTRUMENTS UVIKON 922

Opérateur

RC

FOURNIER 1001793

Highly Confidential  
Subject to  
Protective Order

Mw. 07.97

09

OPERATEUR : ROSSELIN C  
DATE : 01/07/97 ✓  
APPAREIL : GAL091 GAL233 ✓  
LONGUEUR D'ONDE : 290 nm ✓  
CUVE en mm: 2 mm ✓

TITRE : LF178TER comprimés lot2398/01 RG 14Kg - 0,1M 90TPM  
N° CAHIER : LF178ter dissolution n°2 page 088  
FICHIER : n:\commun\glnq\donnbase\LF178ter\dissolution\lot 2398rg01 14 kg 0,1M 90tpm  
ELUANT : LSNa 0,1M  
AGITATION : 90 TPM

masse théorique	160
dosage théorique	160 en mg

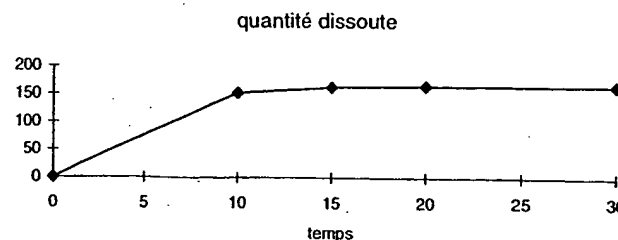
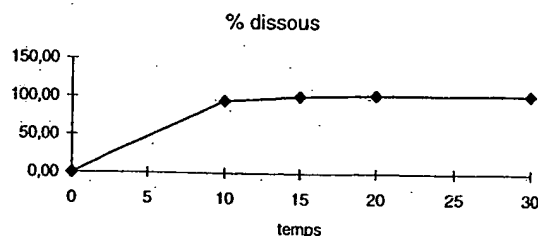
masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/l	0,900
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FOURNIER 1001794

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Protective Order**

[illegible][illegible][illegible]

ML 10.07.97

092  
01.07.97

Dissolution LF178TER comprimés  
lot 2401/01 RG-15Kg  
LSNa 0,1M - 90TPM

1 - Préparation du milieu de dissolution  
cf page 088

2 - Pesée des bbs de dissolution - Balance GALOGS  
1 litre de LSNa 0,1M pèse 1004,0g -

01.07.97 13:49:22  
Code 1

0.00 g  
768.8 g Tare  
0.00 g  
1004.0 g Net ✓  
0.00 g  
1772.8 g Bunt

Code 2

0.00 g  
753.0 g Tare  
0.00 g  
1004.0 g Net ✓  
0.00 g  
1757.0 g Bunt  
RC

Code 3

0.00 g  
760.8 g Tare  
0.00 g  
1004.0 g Net ✓  
0.00 g  
1764.8 g Bunt

Code 4

0.00 g  
698.8 g Tare  
0.00 g  
1004.0 g Net ✓  
0.00 g  
1702.8 g Bunt  
RC

Code 5

0.00 g  
760.5 g Tare  
0.00 g  
1004.0 g Net ✓  
0.00 g  
1764.4 g Bunt

Compteur 0 Re  
Code 6

0.00 g  
767.0 g Tare  
0.00 g  
1004.0 g Net ✓  
0.00 g  
1771.0 g Bunt  
RC

3 - Conditions de dissolution -

Dissolvent Solax AT7 GAL091 -

Température :  $37 \pm 0.5^\circ\text{C}$  RC 01.07.97 RC 01/07/97

Agitation : 90TPM - RC 01.07.97 RC 01/07/97

FOURNIER 1001795

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

# 4. Pesée des comprimés - Balance GAL 205

093

LF178TER  
01.07.97 15:10:03  
Code RG 2401/01-15Kg

0.0 mg  
694.5 mg  
0.0 mg  
698.7 mg  
0.0 mg  
697.4 mg  
0.0 mg  
696.2 mg  
0.0 mg  
699.8 mg  
0.0 mg  
691.1 mg

RL

## 5. Lecture

Sur spectrophotomètre UVIKON 922 GAL 233 en cuve de 2mm de trajet optique à

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER 2401/01RG 15KG LSNA 0.1M 90 TPM

07-01-1997 16:14

Lambda	No.	Valeur_E
290.0	1	-0.0000_1 <i>An / An</i>
290.0	2	-0.0017_1 <i>LSNa / LSNa</i>
290.0	3	-0.0019_1
290.0	4	-0.0026_1
290.0	5	-0.0022_1 <i>10 mm</i>
290.0	6	-0.0022_1
290.0	7	-0.0024_1
290.0	8	-0.0019_1
290.0	9	1.2655_1
290.0	10	1.2605_1
290.0	11	1.2825_1 <i>10 mm</i>
290.0	12	1.2803_1
290.0	13	1.2772_1
290.0	14	1.3170_1
290.0	15	1.4203_1
290.0	16	1.4283_1
290.0	17	1.4229_1 <i>15 mm</i>
290.0	18	1.4227_1
290.0	19	1.4482_1
290.0	20	1.4314_1
290.0	21	1.4444_1
290.0	22	1.4607_1
290.0	23	1.4472_1 <i>20 mm</i>
290.0	24	1.4507_1
290.0	25	1.4773_1
290.0	26	1.4513_1
290.0	27	1.4563_1
290.0	28	1.4665_1
290.0	29	1.4596_1 <i>30 mm</i>
290.0	30	1.4561_1
290.0	31	1.4868_1
290.0	32	1.4664_1

FOURNIER 1001796  
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Subject to  
Protective Order

KONTRON INSTRUMENTS UVIKON 922

Opérateur RL

M 10.07.97

# DISSOLUTION

m:\commun\glng\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 01/07/97 ✓  
APPAREIL : GAL091 GAL233 ✓  
LONGUEUR D'ONDE : 290 nm ✓  
CUVE en mm: 2 mm ✓

TITRE : LF1781ER comprimés lot2401/01 RG 15Kg - 0,1M 90TPM  
N° CAHIER : LF1781er dissolution n°2 page 092  
FICHER : n:\commun\gln\donnbase\lf1781er\dissolution\lot 2401rg01 15 kg 0,1M 90tpm  
ELUANT : LSNa 0,1M  
AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001797

**Highly Confidential  
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Témoin 100mg/l	0,900
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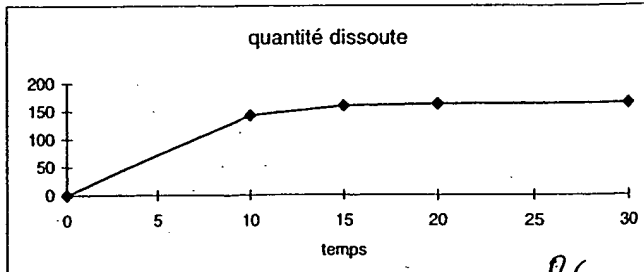
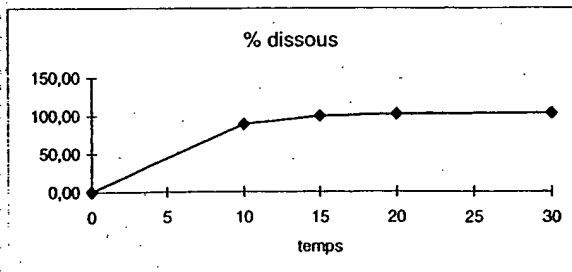
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOLTE

[illegible]

122 10.07.95

01.07.97

Dissolution LF 178TER Comprimés

095

lot 2401/01 RG - 20 Kg

LSNa 0,1M - 90TPM

## 1 - Préparation du milieu de dissolution. Balance GALLOS

01.07.97 Eau purifiée 16:49:51  
0.00 g

642.4 g Tare

0.00 g

2655.9 g Net

0.00 g

3298.3 g Brut

0.00 g

644.5 g Tare

0.00 g

2685.5 g Net

0.00 g

3330.0 g Brut

0.00 g

644.2 g Tare

0.00 g

1942.0 g Net

0.00 g

2586.2 g Brut

RC

Total de l'eau purifiée pesée :

$$2655,9 + 2685,5 + 1942,0 = 7283,4 \text{ g}$$

Pesée de LSNa pour un milieu à 0,1M.  $\text{PH}_{\text{LSNa}}$   
 $= 288,4 \text{ g}$

$$\frac{7283,4}{1000} \times 0,1 \times 288,4 = 210,05 \text{ g}$$

LSNa

01.07.97 17:12:03  
Code AHA 1547

0.00 g

143.22 g Tare

0.00 g

210.06 g Net

0.00 g

353.28 g Brut

RC

## 2 - Pesée des bords de dissolution - Balance GALLOS

1 litre de LSNa 0,1M pèse 1004,0g -

M 10.07.97

FOURNIER 1001798

Highly Confidential  
 Subject to  
 Protective Order



096

01.07.97  
Code

17:46:18

Code

3

Code

5

0.00 g

0.00 g

0.00 g

760.8 g Tare

767.0 g Tare

768.8 g Tare

0.00 g

0.00 g

0.00 g

1004.0 g Net

1004.0 g Net

1004.0 g Net

0.00 g

0.00 g

0.00 g

1764.8 g Bulk

1771.0 g Bulk

1772.8 g Bulk

Code

2

Code

4

Code

6

0.00 g

0.00 g

0.00 g

760.5 g Tare

753.5 g Tare

698.8 g Tare

0.00 g

0.00 g

0.00 g

1004.0 g Net

1004.0 g Net

1004.0 g Net

0.00 g

0.00 g

0.00 g

1764.4 g Bulk

1757.5 g Bulk

1702.8 g Bulk

02.07.97

3 - Conditions de dissolution.

Dissolutese Solax AT7 - GAL091.

Température :  $37^{\circ}\text{C} \pm 0,5^{\circ}\text{C}$  RC 02.07.97 & 02/07/98

Agitation : 90TPM RC 02.07.97 &amp; 02/07/98

4 - Pesée de comprimés GAL 205.LF 178TER  
02.07.97 10:50:26  
Code AG 2401/01-20 kg

0.0 mg

693.8 mg

0.0 mg

695.3 mg

0.0 mg

701.4 mg

0.0 mg

695.7 mg

0.0 mg

698.1 mg

0.0 mg

699.2 mg

FOURNIER 1001799

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M 10.07.97

Sur Spectrophotomètre UVIKON 922 GAL 233 en cuve de  
2 mm de trajet optique à 290 nm

LONGUEUR D ONDE FIXE 290 NM 2 MM.LF178TER 2401/01RG 20KG LSNA 0.1M 90 TPM

07-02-1997 11:34

Lambda	No.	Valeur_E
290.0	1	0.0001_1 <i>A<sub>in</sub>/A<sub>in</sub></i>
290.0	2	-0.0019_1 <i>LSNa/LSNa</i>
290.0	3	-0.0015_1
290.0	4	-0.0012_1
290.0	5	-0.0013_1 <i>0 mm</i>
290.0	6	-0.0012_1
290.0	7	-0.0004_1
290.0	8	-0.0004_1
290.0	9	0.9834_1
290.0	10	0.9881_1
290.0	11	0.9210_1 <i>10 mm</i>
290.0	12	1.0296_1
290.0	13	1.0175_1
290.0	14	1.0399_1
290.0	15	1.3396_1
290.0	16	1.3499_1
290.0	17	1.2943_1 <i>15 mm</i>
290.0	18	1.3697_1
290.0	19	1.3736_1
290.0	20	1.3613_1
290.0	21	1.4372_1
290.0	22	1.4581_1
290.0	23	1.4476_1 <i>20 mm</i>
290.0	24	1.4465_1
290.0	25	1.4601_1
290.0	26	1.4667_1
290.0	27	1.4645_1
290.0	28	1.4877_1
290.0	29	1.4847_1 <i>30 mm</i>
290.0	30	1.4642_1
290.0	31	1.4855_1
290.0	32	1.4813_1

KONTRON INSTRUMENTS UVIKON 922

Opérateur

RL

FOURNIER 1001800

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

## CONDITIONS ET MODÈS OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 02/07/97 ✓  
APPAREIL : GAL091 GAL233 ✓  
LONGUEUR D'ONDE : 290 nm ✓  
CUVE en mm: 2 mm ✓

TITRE : LF178TER comprimés lot2401/01 RG 20Kg - 0,1M 90TPM  
N° CAHIER : LF178ter dissolution n°2 page 095  
FICHER : n:\commun\ng\donnbase\lf178ter\dissolution\lot 2401rg01 20 kg 0,1M 90tpm  
ELUANT : LSNa 0,1M  
AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

**FOURNIER 1001801**

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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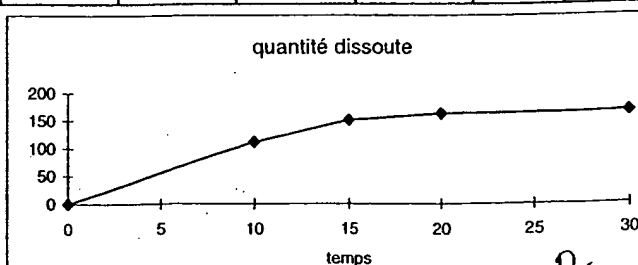
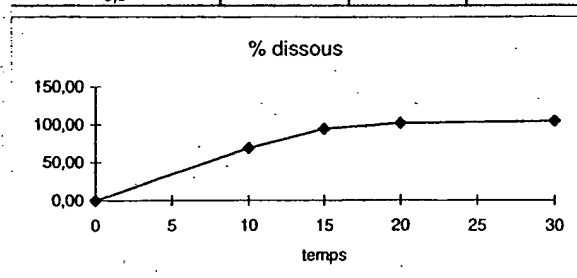
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOLTE

[illegible]

RC  
m 10.07.97

03.07.97

099

Dissolution LF178 compriméslot 2395/01 RG - 14 KgLSNa 0,1M - 30TPM.1 - Préparation du milieu de dissolution - Balance GALL65

Eau purifiée

03.07.97

10:06:14

0.00 g

642.4 g Tare

0.00 g

2475.2 g Net

~~0.01 g~~ Hc

0.00 g

3117.5 g Brut

0.00 g

644.9 g Tare

0.00 g

2495.6 g Net

0.00 g

3140.5 g Brut

0.00 g

645.3 g Tare

0.00 g

2195.7 g Net

0.00 g

2841.0 g Brut

Hc

Total de l'eau purifiée pesée:

$$2475,2 + 2495,6 + 2195,7 = 7166,5g$$

Pesée de LSNa pour un milieu à 0,1M

$$PM_{LSNa} = 288,4g$$

$$\frac{7166,5}{1000} \times 0,1 \times 288,4 = 206,68g$$

LSNa

03.07.97

10:28:54

Code

ARR

1547,

0.00 g

143.22 g Tare

0.00 g

206.68 g Net

0.00 g

349.90 g Brut

Hc

2 - Pesée des bols de dissolution - Balance GALL65

1 litre de LSNa 0,1M pèse 1004,0g -

FOURNIER 1001802

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Protective Order

M 10.07.97

100

03.07.97  
Code

10:48

1

Code

3

5

0.00 g

760.8 g Tare

0.00 g

1004.0 g Net,

0.00 g

1764.8 g Bulk

Code

2

0.00 g

768.8 g Tare

0.00 g

1004.0 g Net,

0.00 g

RC 1772.8 g Bulk

0.00 g

698.8 g Tare

0.00 g

1004.0 g Net,

0.00 g

1702.8 g Bulk

Code

4

0.00 g

767.0 g Tare

0.00 g

1004.0 g Net,

0.00 g

RC 1771.0 g Bulk

0.00 g

760.5 g Tare

0.00 g

1004.0 g Net

0.00 g

1764.4 g Bulk

Code

6

0.00 g

753.0 g Tare

0.00 g

1004.0 g Net,

0.00 g

1757.0 g Bulk

RC

3 - Conditions de dissolution

Dissolutor Solar AT7 - GAC 091

Température: 37°C ± 0,5°C RC 03.07.97 &amp; 03/08/97

Agitation: 50 RPM RC 03.07.97 &amp; 03/08/97

4 - Pesée des comprimés - Balance GAC 205

LF172 TER

03.07.97 12:56:41

Code RC-2395/01-14Kg

0.0 mg

697.2 mg

0.0 mg

699.4 mg

0.0 mg

698.2 mg

0.0 mg

696.8 mg

0.0 mg

697.7 mg

0.0 mg

700.1 mg

RC

FOURNIER 1001803

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Protective Order

M 10.07.97

# 5. Lecture

101

Sur spectrophotomètre UVIKON 922 GAL233 en cuve  
de 2cm de trajet optique à 290 nm.

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER 2395/01RG 14KG LSNA 0.1M 90TPM /

07-03-1997 13:37

Lambda	No.	Valeur_E
290.0	1	0.0004_1 <i>Air Air</i>
290.0	2	-0.0029_1 <i>LSNa LSNa</i>
290.0	3	-0.0027_1
290.0	4	-0.0031_1
290.0	5	-0.0026_1 <i>0 min</i>
290.0	6	-0.0028_1
290.0	7	-0.0026_1
290.0	8	0.0468_1 <i>Cuve mal positionnée - substance</i>
290.0	9	-0.0030_1
290.0	10	1.2689_1
290.0	11	1.2752_1
290.0	12	1.2740_1 <i>10 min</i>
290.0	13	1.2853_1
290.0	14	1.2896_1
290.0	15	1.2808_1
290.0	16	1.3227_1
290.0	17	1.3513_1
290.0	18	1.3111_1 <i>15 min</i>
290.0	19	1.3396_1
290.0	20	1.3437_1
290.0	21	1.3396_1
290.0	22	1.3509_1
290.0	23	1.3681_1
290.0	24	1.3397_1 <i>20 min</i>
290.0	25	1.3570_1
290.0	26	1.3595_1
290.0	27	1.3563_1
290.0	28	1.3615_1
290.0	29	1.4028_1
290.0	30	1.3455_1 <i>20 min</i>
290.0	31	1.3501_1
290.0	32	1.3645_1
290.0	33	1.3646_1

KONTRON INSTRUMENTS UVIKON 922

Opérateur .....

RL

FOURNIER 1001804

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : ROSSELIN C  
DATE : 03/07/97  
APPAREIL : GAL091 GAL233  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2 mm

TITRE : LF178TER comprimés lot2395/01 RG 14Kg - 0,1M 90TPM  
N° CAHIER : LF178ter dissolution n°2 page 099 ✓  
FICHIER : n:\commun\glnq\donnbase\LF178ter\dissolution\lot 2395rg01 14 kg 0,1M 90tpm  
ELUANT : LSNa 0,1M ✓  
AGITATION : 90 TPM ✓

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/l	0,900
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FOURNIER 1001805

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Protective Order**

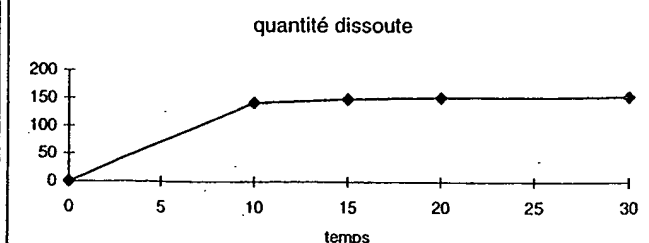
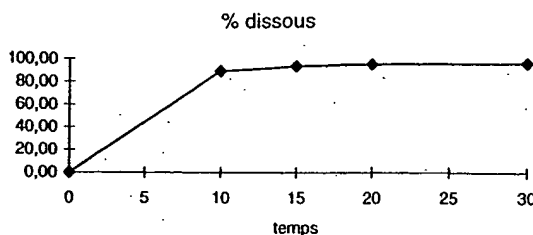
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOUTE

[illegible]

RC

03.07.97

Dissolution LF178 comprimés

103

bt 2395/01 RG - 14kg

LSNa 905M - 120TPH

## 1 - Préparation du milieu de dissolution - Balance GALL065 -

03.07.97 <sup>tempurific</sup> 15:19:01  
0.00 g

642.4 g Tare

0.00 g

2473.2 g Net

0.00 g

3115.6 g Buck

~~0.01 g~~

0.00 g

644.6 g Tare

0.00 g

2492.0 g Net

0.00 g

3136.6 g Buck

0.00 g

644.8 g Tare

0.00 g

1908.3 g Net

0.00 g

2553.1 g Buck

RC

Total de l'eau perifiée pesée -

$$2473,2 + 2492,0 + 1908,3 = 6873,5g$$

Pesée de LSNa pour un milieu à  
905M - PH<sub>LSNa</sub> = 288,4g

$$\frac{6873,5}{1000} \times 905 \times 288,4 = 99,12g$$

LSNa

03.07.97 15:32:11  
Code ARR 1547

0.00 g

143.23 g Tare

0.00 g

99.12 g Net

0.00 g

242.35 g Buck

RC

## 2 - Pesée des bords de dissolution - Balance GALL065 -

1 litre de LSNa 905M pèse 1002,0g -

FOURNIER 1001806  
Highly Confidential  
Subject to  
Protective Order

M 10.07.97



104

Code

1

Code

3

Code

5

0.00 g

760.5 g Tare

0.00 g

1002.0 g Net /

0.00 g

1762.5 g Buft

Code

2

Code

4

Code

6

0.00 g

760.8 g Tare

0.00 g

1002.0 g Net /

0.00 g

1762.8 g Buft  
RC

0.00 g

766.9 g Tare

0.00 g

1002.0 g Net /

0.00 g

1769.0 g Buft  
RC

0.00 g

768.8 g Tare

0.00 g

1002.0 g Net /

0.00 g

1770.8 g Buft

0.00 g

753.0 g Tare

0.00 g

1002.0 g Net /

0.00 g

1755.0 g Buft  
RC3 - Conditions de dissolution

Dissolvest Solar ATF GALO91

Température :  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  - RC 0307.97 - RC 3/7/97

Agitation : 120 TPM - RC 0307.97 - RC 3/7/97

4 - Pesée des comprimés - Balance GAL 205LF 1787  
03.07.97 16:39:47  
Code RC 2395/01-14 kg

0.0 mg

695.6 mg

0.0 mg

695.1 mg

0.0 mg

694.8 mg

0.0 mg

692.4 mg

0.0 mg

699.6 mg

0.0 mg

697.7 mg

RC

FOURNIER 1001807

Highly Confidential  
Subject to  
Protective Order

M 10.07.97

5. Lectures

105

Su Spectrophotomètre UVIKON 922 GAL233. en cuve de 2mm  
de trajet optique à 290 nm.

LONGUEUR D ONDE FIXE 290NM 2MM LF178TER 2395/01RG 14KG LSNA 0.05M 120TPM

07-03-1997 17:26

Lambda	No.	Valeur_E
290.0	1	0.0005_1 <i>Pin</i>
290.0	2	-0.0004_1 <i>LSNa/LSNa</i>
290.0	3	-0.0011_1
290.0	4	-0.0009_1
290.0	5	-0.0011_1 <i>8min</i>
290.0	6	-0.0012_1
290.0	7	-0.0010_1
290.0	8	-0.0012_1
290.0	9	1.1647_1
290.0	10	1.1844_1
290.0	11	1.1991_1 <i>10min</i>
290.0	12	1.1886_1
290.0	13	1.2047_1
290.0	14	1.2093_1
290.0	15	1.2743_1
290.0	16	1.2837_1
290.0	17	1.2998_1 <i>15min</i>
290.0	18	1.2780_1
290.0	19	1.3039_1
290.0	20	1.2946_1
290.0	21	1.3318_1
290.0	22	1.3544_1
290.0	23	1.3671_1 <i>30min</i>
290.0	24	1.3362_1
290.0	25	1.3684_1
290.0	26	1.3386_1

KONTRON INSTRUMENTS UVIKON 922

Opérateur *RC*

pas de prélèvement à  $T = 20$  min, dissolution à reflux.

04.07.97

1. Préparation du milieu de dissolution. Balance OHAUS

FOURNIER 1001808  
Highly Confidential  
Subject to  
Protective Order

M 10.07.97

106

08.07.97

LF178 Ver Comprimés  
 lot RG 2395/01 - 14 kg  
 LSNu 0.05N 120TPN

# 1. Préparation du milieu de dissolution Bal. GAL 065

Pesée de l'eau

EAU

08.07.97 08:59:49  
 0.00 g

229.52 g Taré

0.00 g

2692.0 g Net

0.00 g

2921.5 g Brut

0.00 g

229.58 g Taré

0.00 g

2591.5 g Net

0.00 g

2821.1 g Brut

0.00 g

229.58 g Taré

0.00 g

2716.5 g Net

0.00 g

2946.1 g Brut

o Total de l'eau pesée

$$2692 + 2591,5 + 2716,5 = 8000 \text{ g}$$

o Pesée du LSNu

Pour un milieu à 0,05 N

CPN = 288,4 g

$$\rightarrow \frac{8000}{1000} \times 288,4 \times 0,05 = 115,4 \text{ g}$$

LSNu

08.07.97 09:29:11  
 Code ARR 1547

0.00 g

143.22 g Taré

0.00 g

115.40 g Net

0.00 g

258.62 g Brut

FOURNIER 1001809

Highly Confidential  
 Subject to  
 Protective Order

PC 08.07.97

2. Pesée du milieu de dissolution Bal. GAL 0651 Litre Ligne 005 N  $\rightarrow$  1002,0 g

08.07.97 ID	10:31:57 1	ID	3	ID	5
0.00 g		0.00 g		0.00 g	
760.5 g Tare		767.0 g Tare		753.0 g Tare	
0.00 g		0.00 g		0.00 g	
1002.0 g Net		1002.0 g Net		1002.0 g Net	
0.00 g		0.00 g		0.00 g	
1762.5 g Brut		1769.0 g Brut		1755.0 g Brut	
ID	2	ID	4	ID	6
0.00 g		0.00 g		0.00 g	
768.8 g Tare		698.8 g Tare		760.8 g Tare	
0.00 g		0.00 g		0.00 g	
1002.0 g Net		1002.0 g Net		1002.0 g Net	
0.00 g		0.00 g		0.00 g	
1770.8 g Brut		1700.8 g Brut		1762.8 g Brut	

3. Conditions de dissolution

Dissolubest GAL 091

T° = 37°C  $\pm$  0.5 M 08.07.97 RC 08.07.97

G = 120 TPN M 08.07.97 RC 08.07.97

4. Pesée des comprimés Bal GAL 205

08.07.97 ID	10:12:18 1	ID	3	ID	5	ID	6
0.0 mg		0.0 mg		0.0 mg		0.0 mg	
700.2 mg		697.3 mg		699.1 mg		705.7 mg	
0.0 mg		0.0 mg		0.0 mg		0.0 mg	
700.2 mg		697.6 mg		699.1 mg		705.7 mg	
0.0 mg		0.0 mg		0.0 mg		0.0 mg	
700.2 mg		697.6 mg		699.1 mg		705.7 mg	
0.0 mg		0.0 mg		0.0 mg		0.0 mg	
700.2 mg		697.6 mg		699.1 mg		705.7 mg	

FOURNIER 1001810  
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Protective Order

RC 09.07.97

5. Lecture

Spectrophotomètre GAL 283 - Chronomètre G&amp;H 12h

Cave 2mm - Lecture à 290nm

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.05M 120 TPM

07-08-1997 15:33

Lambda No. Valeur\_E

290.0	1	-0.0003_1
290.0	2	0.0015_1
290.0	3	0.0004_1
290.0	4	-0.0006_1
290.0	5	-0.0001_1
290.0	6	-0.0009_1
290.0	7	1.1805_1
290.0	8	1.1808_1
290.0	9	1.1760_1
290.0	10	1.1825_1
290.0	11	1.1708_1
290.0	12	1.1734_1
290.0	13	1.2874_1
290.0	14	1.2888_1
290.0	15	1.2761_1
290.0	16	1.2910_1
290.0	17	1.2900_1
290.0	18	1.2763_1
290.0	19	1.3266_1
290.0	20	1.3192_1
290.0	21	1.3080_1
290.0	22	1.3297_1
290.0	23	1.3287_1
290.0	24	1.3173_1
290.0	25	1.3431_1
290.0	26	1.3381_1
290.0	27	1.3252_1
290.0	28	1.3433_1
290.0	29	1.3469_1
290.0	30	1.3380_1
290.0	31	1.3519_1
290.0	32	1.3419_1
290.0	33	1.3397_1
290.0	34	1.3514_1
290.0	35	1.3580_1
290.0	36	1.3507_1
290.0	37	1.3529_1
290.0	38	1.3493_1
290.0	39	1.3434_1
290.0	40	1.3581_1
290.0	41	1.3589_1
290.0	42	1.3470_1

290.0 1 -0.0000\_1  
 290.0 2 0.0002\_1

① Autogéro Air/Air

② LSNa/LSNa

FOURNIER 1001811

Highly Confidential  
 Subject to  
 Protective Order

AC0307 97

## DISSOLUTION

m:\commun\glng\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 08/07/97 ✓  
APPAREIL : GAL 233 GAL 091 ✓  
LONGUEUR D'ONDE : 290 nm ✓  
CUVE en mm: 2 ✓

TITRE : LF 178 TER RG 2395/01 à 14 KG  
N° CAHIER : LF 178 TER n°2 p 106  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\ot 2395RG01 14 kg 0,05M 120tpm  
ELUANT : LSNa 0,05 M  
AGITATION : 120 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/l	0,900
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**FOURNIER 1001812**

**Highly Confidential  
Subject to  
Protective Order**

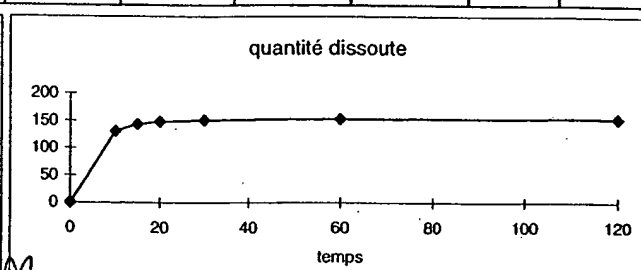
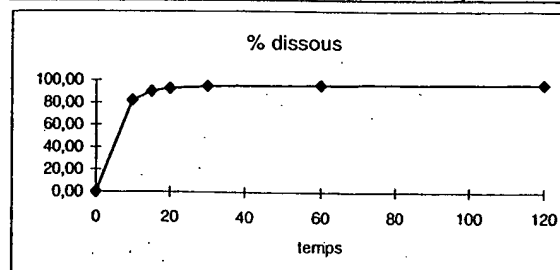
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOLTE

[illegible]

RC 09.07.97

110 09.07.97

LF 178 ter Compaines  
 Lot RG 2385/01. 14 kg.  
 LsNa 0025N - 75 TPN.

1. Préparation du milieu de dissolution. Bal CAL 605

Pesée de l'eau

EAU

09.07.97 09:54:38

0.00 g

229.57 g Tare

0.00 g

2613.5 g Net

0.00 g

2843.1 g Brut

0.00 g

229.57 g Tare

0.00 g

2917.6 g Net

0.00 g

3147.2 g Brut

0.00 g

229.57 g Tare

0.00 g

2475.7 g Net

0.00 g

2705.3 g Brut

M

o Quantité d'eau pesée :

$$2613,5 + 2917,6 + 2475,7 = 8006,8 \text{ g}$$

o Pesée du LsNa

pour un milieu à 0,025N

(CPN 288,4 g)

$$\rightarrow \frac{8006,8}{1000} \times 288,4 \times 0,025 = 57,7 \text{ g}$$

LsNa

09.07.97 10:10:32

Code ARR 1547

0.00 g

143.45 g Tare

0.00 g

57.70 g Net

0.00 g

201.15 g Brut

M

FOURNIER 1001813  
 Highly Confidential  
 Subject to  
 Protective Order

RL09.07.97

2. Pesée du milieu de dissolution Bal. GAL 065 111

1 Lit L3Na 0.025N → 1001.0 g.

09.07.97 ID	10:48:36 1	ID	3	ID	5
0.00 g		0.00 g		0.00 g	
768.8 g Tax		698.8 g Tax		753.0 g Tax	
0.00 g		0.00 g		0.00 g	
1001.0 g Net		1001.0 g Net		1001.0 g Net	
0.00 g		0.00 g		0.00 g	
1769.8 g Brut		1699.8 g Brut		1754.0 g Brut	
ID	2	ID	4	ID	6
0.00 g		0.00 g		0.00 g	
760.5 g Tax		767.0 g Tax		760.8 g Tax	
0.00 g		0.00 g		0.00 g	
1001.0 g Net		1001.0 g Net		1001.0 g Net	
0.00 g		0.00 g		0.00 g	
1761.5 g Brut		1768.0 g Brut		1761.8 g Brut	

3. Pesée des comprimés Bal. GAL 205

LF 178 bar  
09.07.97 10:46:43  
Code RG 2395.01-14 kg.

ID	1	ID	2	ID	3	ID	4	ID	5	ID	6
0.0 mg		0.0 mg		0.0 mg		0.0 mg		0.0 mg		0.0 mg	
693.2 mg		698.0 mg		697.3 mg		697.4 mg		697.5 mg		695.2 mg	

4. Conditions de dissolution  
Dissolvest GAL 081

T: 37°C ± 0.5  
O: 75 TPN

FOURNIER 1001814  
Highly Confidential  
Subject to  
Protective Order

09/07/97 M 04.07.97  
09/07/97 M 04.07.97

09.07.97



5. lecture

Spectrophotométrie GAZ 233 - Chronométré GAZ 124  
Cave 2mm - 290nm

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.025M 75 TPM

07-09-1997 15:17

Lambda No. Valeur\_E

290.0	1	0.0010_1
290.0	2	0.0005_1
290.0	3	0.0006_1
290.0	4	0.0007_1
290.0	5	0.0003_1
290.0	6	0.0004_1
290.0	7	0.9210_1
290.0	8	0.9154_1
290.0	9	0.9103_1
290.0	10	0.9113_1
290.0	11	0.9063_1
290.0	12	0.9098_1
290.0	13	1.0912_1
290.0	14	1.1013_1
290.0	15	1.0968_1
290.0	16	1.0826_1
290.0	17	1.0832_1
290.0	18	1.0792_1
290.0	19	1.1735_1
290.0	20	1.1761_1
290.0	21	1.1763_1
290.0	22	1.1634_1
290.0	23	1.1697_1
290.0	24	1.1688_1
290.0	25	1.2453_1
290.0	26	1.2539_1
290.0	27	1.2517_1
290.0	28	1.2389_1
290.0	29	1.2471_1
290.0	30	1.2452_1
290.0	31	1.2704_1
290.0	32	1.2774_1
290.0	33	1.2783_1
290.0	34	1.2669_1
290.0	35	1.2803_1
290.0	36	1.2711_1
290.0	37	1.2850_1
290.0	38	1.2912_1
290.0	39	1.2948_1
290.0	40	1.2826_1
290.0	41	1.2919_1
290.0	42	1.2856_1
290.0	43	1.2923_1
290.0	44	1.2986_1
290.0	45	1.3045_1
290.0	46	1.2900_1
290.0	47	1.2933_1
290.0	48	1.2934_1
290.0	49	1.3125_1
290.0	50	1.3130_1
290.0	51	1.3248_1
290.0	52	1.3105_1
290.0	53	1.3107_1
290.0	54	1.3181_1

① 290.0 1 -0.0000\_1  
② 290.0 2 -0.0004\_1

① Autozero Air / Air

② LSNa / LSNa

FOURNIER 1001815  
Highly Confidential  
Subject to  
Protective Order

AC 0907 97

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 09/07/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2

TITRE : LF 178 TER RG 2395/01 à 14 KG LSNa 0,025M 75 tpm  
 N° CAHIER : LF 178 TER n°2 p 110  
 FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot 2395RG01 14 kg 0,025M 75tpm  
 ELUANT : LSNa 0,025 M  
 AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/ 0,900

FOURNIER 1001816

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 Subject to  
 Protective Order

## SUIVI DE LA DISSOLUTION

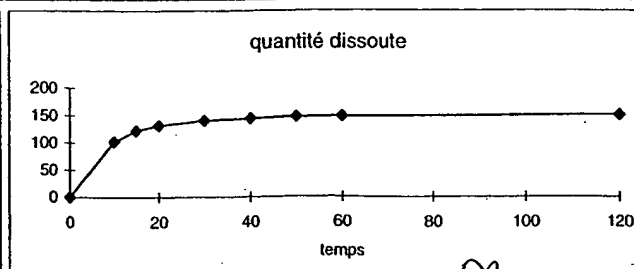
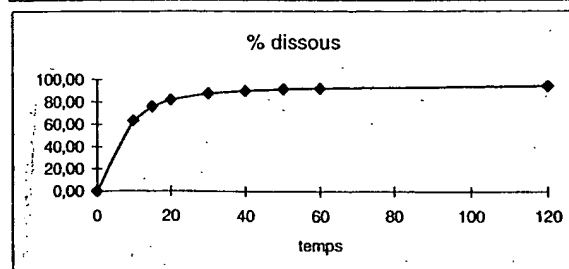
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,921	0,915	0,91	0,911	0,906	0,91
15	1000	1,091	1,101	1,097	1,083	1,083	1,079
20	1000	1,174	1,176	1,176	1,163	1,17	1,169
30	1000	1,246	1,254	1,252	1,239	1,247	1,245
40	1000	1,27	1,277	1,278	1,267	1,28	1,271
50	1000	1,285	1,291	1,295	1,283	1,292	1,286
60	1000	1,292	1,299	1,305	1,29	1,293	1,293
120	1000	1,313	1,313	1,325	1,311	1,311	1,318

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	63,34	63,96	63,54	63,19	63,26	62,92	63,19	0,36	0,57
15,0	75,94	76,08	76,78	76,50	75,52	75,52	75,25	0,61	0,80
20,0	82,04	82,23	82,37	82,36	81,46	81,94	81,87	0,35	0,43
30,0	87,71	87,63	88,19	88,05	87,14	87,69	87,55	0,38	0,43
40,0	90,00	89,73	90,22	90,29	89,51	90,42	89,79	0,36	0,40
50,0	91,47	91,22	91,64	91,91	91,06	91,70	91,28	0,33	0,36
60,0	92,38	92,15	92,64	93,06	92,00	92,21	92,21	0,40	0,43
120,0	94,21	94,06	94,07	94,90	93,90	93,91	94,39	0,38	0,41
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	101,35	102,33	101,67	101,11	101,22	100,67	101,11	0,58	0,57
15,0	121,51	121,73	122,84	122,39	120,84	120,84	120,39	0,98	0,80
20,0	131,26	131,56	131,79	131,78	130,33	131,11	130,99	0,57	0,43
30,0	140,34	140,21	141,11	140,88	139,42	140,31	140,09	0,60	0,43
40,0	143,99	143,57	144,36	144,46	143,22	144,67	143,67	0,58	0,40
50,0	146,35	145,95	146,62	147,06	145,70	146,71	146,04	0,53	0,36
60,0	147,80	147,44	148,23	148,89	147,19	147,54	147,53	0,64	0,43
120,0	150,73	150,49	150,51	151,84	150,24	150,26	151,03	0,61	0,41
0,0									
0,0									
0,0									
0,0									



RC09.07.97

114

17.07.97

LF ABter Comprimés

Lot C0187 - 160 mg

LSNce Q<sub>AT</sub> - 50 TPN

0,025N

1. Préparation du milieu de dissolution Bal. GAT ces

Eau.

17.07.97 15:45:26

0.00 g

448.35 g Tare

0.00 g

3507.7 g Net

0.00 g

3956.0 g Brct

0.00 g

449.72 g Tare

0.00 g

3539.7 g Net

0.00 g

3989.4 g Brct

0.00 g

449.96 g Tare

0.00 g

3614.7 g Net

0.00 g

4064.6 g Brct

0.00 g Tare

449.68 g

0.00 g

2107.9 g Net

0.00 g

2557.5 g Brct

M

Pesée de l'eau

o quantité d'eau pesée

$$3507,7 + 3539,7 + 3614,7 + 2107,9 = 12770$$

Pour un milieu à 0,025N

CPN<sub>LSNce</sub> (288,4g) →

$$\frac{12770}{1000} \times 288,4 \times 0,025 = 92,1g$$

17.07.97 16:01:50  
Code ARR 1547

LSNce

0.00 g

143.22 g Tare

0.00 g

92.11 g M

92.11 g Net

0.00 g

235.34 g Brct

M

FOURNIER 1001817

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Protective Order

PR 22.01.99

## 2. Remplissage des b<sup>ts</sup> de dissolution bal GAL 06S 115

1161 1811 070511 pese 1001.0g.

17.07.97 16:22:01

1 0.00 g  
760.9 g Tax  
0.00 g  
1001.0 g Net  
0.00 g  
1761.9 g Brct

3 0.00 g  
767.0 g Taxe  
0.00 g  
1001.0 g Net  
0.00 g  
1768.0 g Brct

5 0.00 g  
768.8 g Tax  
0.00 g  
1001.1 g Net  
0.00 g  
1769.9 g Brct

2 0.00 g  
753.0 g Tax  
0.00 g  
1001.0 g Net  
0.00 g  
1754.0 g Brct M

4 0.00 g  
698.8 g Tax  
0.00 g  
1001.0 g Net  
0.00 g  
1699.9 g Brct M

6 0.00 g  
760.5 g Tax  
0.00 g  
1001.0 g Net  
0.00 g  
1761.5 g Brct M

## 3. Pesée des comprimés Bal. GAL 06S

IRPT 160mg  
17.07.97 16:18:17  
Code 1401 C 0197

1 ID

0.0 mg  
712.3 mg

2 ID

0.0 mg  
724.3 mg

3 ID

0.0 mg  
718.4 mg

4 ID

0.0 mg  
719.4 mg

5 ID

0.0 mg  
717.1 mg

6 ID

0.0 mg  
709.9 mg

## h. Conditions

Produit GAL 081

T = 37° C ± 0.5 RC 18.07.97 M 18.07.97

Q = 50 TPN RC 18.07.97 M 18.07.97

FOURNIER 1001818

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Subject to  
Protective Order

RC 22.07.97

116 S. Leche

Spectrophotométrie GAL 233 - Chromométrie GAL 233

LF 178 TER LOT C0197 0.025M 50TPM

07-18-1997 10:35

A2

Lambda No. Valeur\_E

290.0	1	0.0000_1
290.0	2	0.0007_1
290.0	3	0.0016_1
290.0	4	0.0026_1
290.0	5	0.0023_1
290.0	6	0.0039_1
290.0	7	0.0024_1
290.0	8	0.0018_1
290.0	9	0.7371_1
290.0	10	0.6970_1
290.0	11	0.7902_1
290.0	12	0.8261_1
290.0	13	0.7324_1
290.0	14	0.7873_1
290.0	15	1.0044_1
290.0	16	0.9964_1
290.0	17	1.0403_1
290.0	18	1.0540_1
290.0	19	1.0167_1
290.0	20	1.0546_1
290.0	21	1.1534_1
290.0	22	1.1590_1
290.0	23	1.1729_1
290.0	24	1.1795_1
290.0	25	1.1691_1
290.0	26	1.1971_1
290.0	27	1.2695_1
290.0	28	1.2789_1
290.0	29	1.2841_1
290.0	30	1.2787_1
290.0	31	1.2862_1
290.0	32	1.3077_1
290.0	33	1.3218_1
290.0	34	1.3335_1
290.0	35	1.3380_1
290.0	36	1.3225_1
290.0	37	1.3410_1
290.0	38	1.3497_1
290.0	39	1.3502_1
290.0	40	1.3602_1
290.0	41	1.3656_1
290.0	42	1.3427_1
290.0	43	1.3662_1
290.0	44	1.3779_1
290.0	45	1.3668_1
290.0	46	1.3783_1
290.0	47	1.3868_1
290.0	48	1.3581_1
290.0	49	1.3810_1
290.0	50	1.3854_1
290.0	51	1.4018_1
290.0	52	1.4052_1
290.0	53	1.4233_1
290.0	54	1.3928_1
290.0	55	1.4169_1
290.0	56	1.4100_1

Aubergine Air/Air  
V202 / V202

FOURNIER 1001819

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Subject to  
Protective Order

V202 2207 97

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 18/07/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2

TITRE : LF 178 TER lot C0197 LSNa 0,025M 50 tpm  
 N° CAHIER : LF 178 TER n°2 p 114  
 FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot C0197 0,025M 50tpm  
 ELUANT : LSNa 0,025 M  
 AGITATION : 50 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Témoins 100mg/l 0,900

FOURNIER 1001820  
 Highly Confidential  
 Subject to  
 Protective Order

## SUIVI DE LA DISSOLUTION

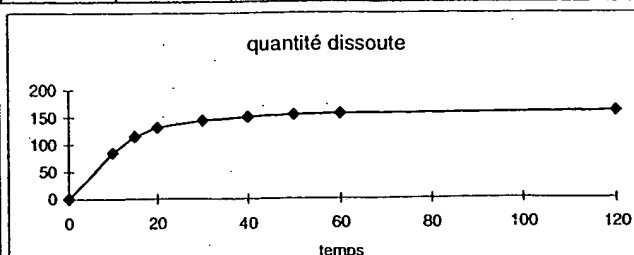
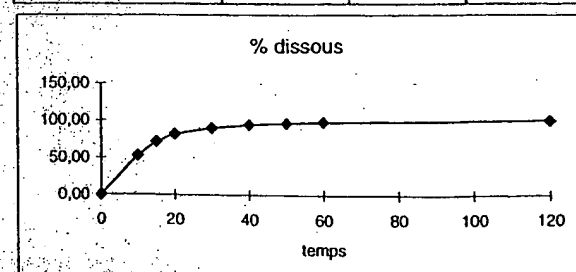
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,737	0,697	0,79	0,826	0,732	0,787
15	1000	1,004	0,996	1,04	1,054	1,017	1,055
20	1000	1,153	1,159	1,173	1,18	1,169	1,197
30	1000	1,27	1,279	1,284	1,279	1,286	1,308
40	1000	1,322	1,334	1,338	1,323	1,341	1,35
50	1000	1,35	1,36	1,366	1,343	1,366	1,378
60	1000	1,367	1,378	1,387	1,358	1,381	1,385
120	1000	1,402	1,405	1,423	1,393	1,417	1,41

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	52,88	51,18	48,40	54,86	57,36	50,83	54,65	3,29	6,23
15,0	71,63	69,98	69,41	72,50	73,48	70,88	73,54	1,79	2,50
20,0	82,00	80,67	81,07	82,09	82,60	81,79	83,76	1,11	1,35
30,0	90,22	89,20	89,81	90,21	89,88	90,32	91,89	0,91	1,01
40,0	94,16	93,25	94,07	94,41	93,38	94,58	95,26	0,76	0,81
50,0	96,42	95,65	96,34	96,81	95,23	96,79	97,67	0,88	0,91
60,0	97,97	97,30	98,06	98,75	96,74	98,30	98,64	0,79	0,81
120,0	100,69	100,21	100,42	101,73	99,64	101,28	100,85	0,76	0,75
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	84,61	81,89	77,44	87,78	91,78	81,33	87,44	5,27	6,23
15,0	114,61	111,97	111,05	115,99	117,57	113,41	117,66	2,87	2,50
20,0	131,20	129,08	129,72	131,35	132,16	130,86	134,02	1,77	1,35
30,0	144,35	142,72	143,70	144,34	143,81	144,51	147,02	1,45	1,01
40,0	150,65	149,20	150,52	151,05	149,41	151,34	152,42	1,22	0,81
50,0	154,27	153,05	154,15	154,90	152,37	154,86	156,28	1,41	0,91
60,0	156,74	155,69	156,90	158,00	154,78	157,28	157,82	1,27	0,81
120,0	161,10	160,34	160,67	162,77	159,42	162,05	161,37	1,21	0,75
0,0									
0,0									
0,0									
0,0									



M

BL 23.02.97

118

18.07.97

Dissolution LF 176ter

lot C 0197 - 160 mg

0,025 N - 30 TPN

1. Préparation du milieu de dissolution

Voir page 114

2. Remplissage des bts de dissolution bal. GAL 06S  
Mise de 18Nce 0,025 N pese 1001,0g.

18.07.97 11:03:02

1 0.00 g  
767.3 g Tare  
0.00 g  
1001.1 g Net  
0.00 g  
1768.4 g Brck

3 0.00 g  
761.2 g Tare  
0.00 g  
1001.0 g Net  
0.00 g  
1762.2 g Brck

5 0.00 g  
760.8 g Tare  
0.00 g  
1001.0 g Net  
0.00 g  
1761.8 g Brck

2 0.00 g  
769.1 g Tare  
0.00 g  
1001.0 g Net  
0.00 g  
1770.1 g Brck M

4 0.00 g  
753.4 g Tare  
0.00 g  
1001.0 g Net  
0.00 g  
1754.4 g Brck M

6 0.00 g  
699.1 g Tare  
0.00 g  
1001.0 g Net  
0.00 g  
1700.1 g Brck M

3. Pesée des comprimés Bal. GAL 20S

FOURNIER 1001821

LF 176ter

18.07.97 12:27:27  
Code lot: C 0197

ID 1

0.0 mg

722.4 mg

ID 2

0.0 mg

721.2 mg

ID 3

0.0 mg

699.2 mg

ID 4

0.0 mg

705.5 mg

ID 5

0.0 mg

711.9 mg

ID 6

0.0 mg

712.2 mg

Highly Confidential  
Subject to  
Protective Order

RC-22.07.97 M

# h. Conditions

Dissectest GAL 091

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5$

$\phi = 90^{\circ}$  TPN

M. B. G. A. T. /  
18/07/97  
M. B. G. A. T. /  
18/07/97

LF 178 TER LOT C0197 0.025M 90TPM

07-18-1997 14:16

Lambda No. Valeur\_E

290.0	1	0.0000_1
290.0	2	0.0007_1
290.0	3	0.0020_1
290.0	4	0.0020_1
290.0	5	0.0014_1
290.0	6	0.0012_1
290.0	7	0.0022_1
290.0	8	0.0010_1
290.0	9	0.9608_1
290.0	10	0.8565_1
290.0	11	0.7551_1
290.0	12	0.7711_1
290.0	13	0.9355_1
290.0	14	0.9723_1
290.0	15	1.1790_1
290.0	16	1.1223_1
290.0	17	1.0645_1
290.0	18	1.0810_1
290.0	19	1.1657_1
290.0	20	1.1714_1
290.0	21	1.2838_1
290.0	22	1.2463_1
290.0	23	1.2074_1
290.0	24	1.2238_1
290.0	25	1.2734_1
290.0	26	1.2626_1
290.0	27	1.3727_1
290.0	28	1.3500_1
290.0	29	1.3170_1
290.0	30	1.3355_1
290.0	31	1.3591_1
290.0	32	1.3471_1
290.0	33	1.4115_1
290.0	34	1.3832_1
290.0	35	1.3602_1
290.0	36	1.3683_1
290.0	37	1.3912_1
290.0	38	1.3783_1
290.0	39	1.4216_1
290.0	40	1.4021_1
290.0	41	1.3742_1
290.0	42	1.3913_1
290.0	43	1.4020_1
290.0	44	1.3902_1
290.0	45	1.4293_1
290.0	46	1.4135_1
290.0	47	1.3848_1
290.0	48	1.3878_1
290.0	49	1.4064_1
290.0	50	1.3986_1

## S. Lecture

Spectro GAL 233

Chronometre GAL RL

FOURNIER 1001822

Highly Confidential  
Subject to  
Protective Order

Re 22.07.97



m:\commun\qlng\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 18/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2

TITRE : LF 178 TER cot C0197 LSNa 0,025M 90 tpm  
N° CAHIER : LF 178 TER n°2 p 118  
FICHIER : M:\commun\glq\donnbase\lf178ter\dissolution\cot C0197 0,025M 90tpm  
ELUANT : LSNa 0,025 M  
AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001823

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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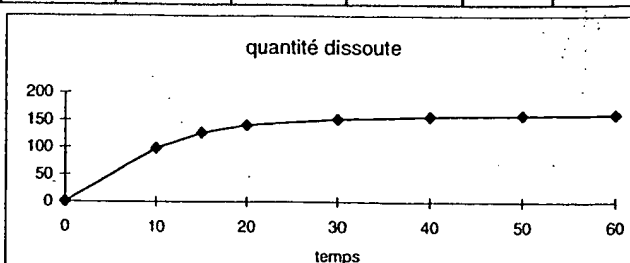
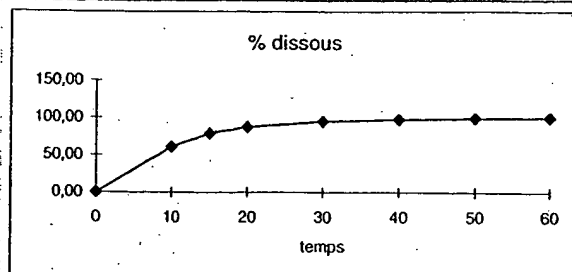
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOLTE

[illegible]

V3 (22.0) 99

21.07.97

Dissolution. LF 178 ter

121

ldr CPT 157. 160 mg.0.025N - 9.120TPN  
M1. Preparation du milieu de dissolution Bal GAL 065

21.07.97 07:58:12

EAU 0.00 g

448.25 g Tar

0.00 g

3548.7 g Net

0.00 g

3996.9 g Brut

0.00 g

449.38 g Tar

0.00 g

3497.1 g Net

0.00 g

3946.4 g Brut

0.00 g

449.43 g Tar

0.00 g

3522.6 g Net

0.00 g

3972.1 g Brut

0.00 g

449.68 g Tar

0.00 g

2549.2 g Net

0.00 g

2998.9 g Brut

M

o Quantité d'eau pesée:

$$3548.7 + 3497.1 + 3522.6 + 2549.2 = 13117.6 \text{ g}$$

Pour un milieu à 0.025N (PN<sub>net</sub> = 288.4g)

$$\frac{13117.6}{1000} \times 288.4 \times 0.025 = 94.6 \text{ g}$$

LSN<sub>net</sub> ARR 1547

21.07.97 08:17:03

0.00 g

143.22 g Tar

0.00 g

94.60 g Net

0.00 g

237.83 g Brut

M

FOURNIER 1001824

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Subject to  
Protective Order

22.07.97

122

2. Remplissage des bds de dissolution Bal GAL 065  
 1 litre L90u 0.025N pese 1001.0g.

21.07.97 08:55:18

1 0.00 g  
 767.2 g Tare  
 0.00 g  
 1001.0 g Net  
 0.00 g  
 1768.2 g Brut

2 0.00 g  
 760.8 g Tare  
 0.00 g  
 1001.0 g Net  
 0.00 g  
 1761.8 g Brut M

3 0.00 g  
 760.5 g Tare  
 0.00 g  
 1001.0 g Net  
 0.00 g  
 1761.5 g Brut

4 0.00 g  
 760.8 g Tare  
 0.00 g  
 1001.0 g Net  
 0.00 g  
 1769.8 g Brut M

5 0.00 g  
 753.0 g Tare  
 0.00 g  
 1001.0 g Net  
 0.00 g  
 1754.0 g Brut

6 0.00 g  
 698.8 g Tare  
 0.00 g  
 1001.0 g Net  
 0.00 g  
 1699.8 g Brut M

3. Pesée des comprimés. Bal GAL 205

LF Abter

21.07.97 08:47:39

Code lot: C 0197

ID 1

0.0 mg

714.4 mg

ID 2

0.0 mg

715.3 mg

ID 3

0.0 mg

716.7 mg

ID 4

0.0 mg

714.8 mg

ID 5

0.0 mg

718.6 mg

ID 6

0.0 mg

704.3 mg

4. Conditions

Dispositif GAL 235 M 091

$T^{\circ} = 37^{\circ}C \pm 0.5$  R-21/1/97 M 21.07.97

$Q = 120$  TPN R-21/1/97 M 21.07.97

RC22.07.97

FOURNIER 1001825

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S. Lecture

Spectrophotométre GAL 233 - Chronométre GAL 22

LF 178 TER LOT C0197 LSNA 0.025 M 120 TPM

07-21-1997 11:02

Lambda No. Valeur\_E

290.0	1	0.0000_1	→ Autozero Au / Mer
290.0	2	0.0007_1	→ LSNa / LSNa
290.0	3	0.0015_1	
290.0	4	0.0012_1	
290.0	5	0.0023_1	
290.0	6	0.0008_1	
290.0	7	0.0015_1	
290.0	8	0.0010_1	
290.0	9	1.0002_1	
290.0	10	1.0016_1	
290.0	11	0.9118_1	
290.0	12	0.9798_1	
290.0	13	1.0057_1	
290.0	14	0.9144_1	
290.0	15	1.2003_1	
290.0	16	1.2040_1	
290.0	17	1.1608_1	
290.0	18	1.1826_1	
290.0	19	1.1932_1	
290.0	20	1.1604_1	
290.0	21	1.2969_1	
290.0	22	1.2934_1	
290.0	23	1.2777_1	
290.0	24	1.2765_1	
290.0	25	1.2913_1	
290.0	26	1.2718_1	
290.0	27	1.3701_1	
290.0	28	1.3678_1	
290.0	29	1.3747_1	
290.0	30	1.3582_1	
290.0	31	1.3689_1	
290.0	32	1.3555_1	
290.0	33	1.3999_1	
290.0	34	1.3949_1	
290.0	35	1.4103_1	
290.0	36	1.3835_1	
290.0	37	1.3996_1	
290.0	38	1.3868_1	
290.0	39	1.4146_1	
290.0	40	1.4061_1	
290.0	41	1.4245_1	
290.0	42	1.3931_1	
290.0	43	1.4078_1	
290.0	44	1.4063_1	
290.0	45	1.4194_1	
290.0	46	1.4075_1	
290.0	47	1.4310_1	
290.0	48	1.4011_1	
290.0	49	1.4112_1	
290.0	50	1.4119_1	

Résultats traités  
Page 127.FOURNIER 1001826  
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Subject to  
Protective Order

RC 22.07.97

124

21.07.97

Dissolution LF 178 Ten

Ltr RG 2395/01 1h kg

LNa 0,025N. 50 TPN

1. Préparation du milieu de dissolution

Voir page 121

2. Remplissage des bés de dissolution Bal GAL 065

Ltr LNa 0,025N pèse 1001,0 g

21.07.97 11:11:14

1 767.6 g Taxe

0.00 g

1001.0 g Net

0.00 g

1768.6 g Brut

2 0.00 g

761.2 g Taxe

0.00 g

1001.0 g Net

0.00 g

1762.2 g Brut m

3 0.00 g

761.4 g Taxe

0.00 g

1001.0 g Net

0.00 g

1762.4 g Brut

4 0.00 g

753.6 g Taxe

~~-0.01 g M~~

0.00 g

1001.0 g Net

0.00 g

1754.6 g Brut m

5 0.00 g

699.3 g Taxe

~~-0.01 g M~~

0.00 g

1001.0 g Net

0.00 g

1700.3 g Brut

6 0.00 g

769.3 g Taxe

0.00 g

1001.0 g Net

0.00 g

1770.4 g Brut m

3. Pesée des compures Bal. GAL 205

FOURNIER 1001827

Highly Confidential  
Subject to  
Protective OrderLF 178 Ten  
21.07.97 12:38:19  
Code RG 2395/01-14 kg.

1

0.0 mg

694.5 mg

2

0.0 mg

696.6 mg

3

0.0 mg

697.3 mg

4

0.0 mg

702.6 mg

5

0.0 mg

697.9 mg

6

0.0 mg

697.5 mg

m

Pic 22.07.97

h. Conditions

125

Resolved GAL 081

T: 37°C ± 0.5 AG 21/7/97 M 21.07.97

O: 50 TPM AG 21/7/97 M 21.07.97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.025 M 50 TPM

07-21-1997 14:57

S. lecture

Spectrophotométrie GAL 083

Chromométrie GAL 082

Lambda	No.	Valeur_E
290.0	1	-0.0000_1
290.0	2	0.0007_1
290.0	3	0.0011_1
290.0	4	0.0010_1
290.0	5	0.0009_1
290.0	6	0.0016_1
290.0	7	0.0017_1
290.0	8	0.0010_1
290.0	9	0.7969_1
290.0	10	0.7814_1
290.0	11	0.7834_1
290.0	12	0.7716_1
290.0	13	0.7999_1
290.0	14	0.7725_1
290.0	15	0.9906_1
290.0	16	0.9809_1
290.0	17	0.9887_1
290.0	18	0.9941_1
290.0	19	1.0035_1
290.0	20	0.9833_1
290.0	21	1.0910_1
290.0	22	1.0909_1
290.0	23	1.0910_1
290.0	24	1.1049_1
290.0	25	1.1027_1
290.0	26	1.0912_1
290.0	27	1.1816_1
290.0	28	1.1881_1
290.0	29	1.1871_1
290.0	30	1.2090_1
290.0	31	1.2015_1
290.0	32	1.1920_1

FOURNIER 1001828

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PC 22.07.97

O- 50 TPM

AR 21/7/97 M 21 07 CA

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.025 M 50 TPM

07-21-1997 14:57

Lambda No. Valeur\_E

S. lecture

Spectrophotometer GAL 183

Chronometre GAL 182

290.0	1	-0.0000_1
290.0	2	0.0007_1
290.0	3	0.0011_1
290.0	4	0.0010_1
290.0	5	0.0009_1
290.0	6	0.0016_1
290.0	7	0.0017_1
290.0	8	0.0010_1
290.0	9	0.7969_1
290.0	10	0.7814_1
290.0	11	0.7834_1
290.0	12	0.7716_1
290.0	13	0.7999_1
290.0	14	0.7725_1
290.0	15	0.9906_1
290.0	16	0.9809_1
290.0	17	0.9887_1
290.0	18	0.9941_1
290.0	19	1.0035_1
290.0	20	0.9833_1
290.0	21	1.0910_1
290.0	22	1.0909_1
290.0	23	1.0910_1
290.0	24	1.1049_1
290.0	25	1.1027_1
290.0	26	1.0912_1
290.0	27	1.1816_1
290.0	28	1.1881_1
290.0	29	1.1871_1
290.0	30	1.2090_1
290.0	31	1.2015_1
290.0	32	1.1920_1
290.0	33	1.2239_1
290.0	34	1.2239_1
290.0	35	1.2297_1
290.0	36	1.2496_1
290.0	37	1.2451_1
290.0	38	1.2308_1
290.0	39	1.2502_1
290.0	40	1.2545_1
290.0	41	1.2568_1
290.0	42	1.2793_1
290.0	43	1.2710_1
290.0	44	1.2560_1
290.0	45	1.2603_1
290.0	46	1.2696_1
290.0	47	1.2744_1
290.0	48	1.2895_1
290.0	49	1.2800_1
290.0	50	1.2703_1
290.0	51	1.3128_1
290.0	52	1.3152_1
290.0	53	1.3244_1
290.0	54	1.3489_1
290.0	55	1.3275_1
290.0	56	1.3175_1

RC 22.07.97

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Protective Order

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 21/07/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVES en mm: 2

TITRE : LF 178 TER lot RG 2395/01 14 kg LSNa 0,025M 50 tpm  
 N° CAHIER : LF 178 TER n°2 p 124  
 FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot RG 2395/01 14 kg 0,025M 50tpm  
 ELUANT : LSNa 0,025 M  
 AGITATION : 50 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001830

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 Subject to  
 Protective Order

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,797	0,781	0,783	0,772	0,8	0,773
15	1000	0,991	0,981	0,989	0,994	1,004	0,983
20	1000	1,091	1,091	1,091	1,105	1,103	1,091
30	1000	1,187	1,188	1,187	1,209	1,202	1,192
40	1000	1,224	1,224	1,23	1,25	1,245	1,231
50	1000	1,25	1,255	1,257	1,279	1,271	1,256
60	1000	1,26	1,27	1,274	1,29	1,28	1,27
120	1000	1,313	1,315	1,324	1,349	1,328	1,318

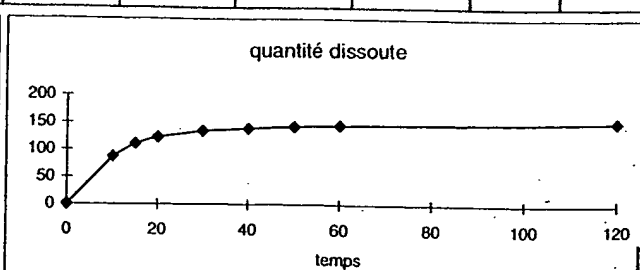
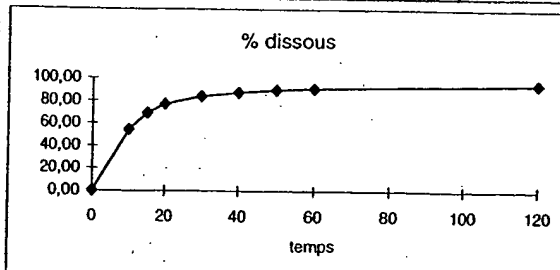
## RESULTATS EN % DISSOUS

ok sans incidence significative sur les résultats AC 22.07.97

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0	0	0
10,0	54,47	55,35	54,24	54,38	53,61	55,56	53,68	0,82	1,51
15,0	69,05	69,10	68,40	68,95	69,30	70,00	68,53	0,58	0,84
20,0	76,68	76,38	76,38	76,38	77,35	77,22	76,37	0,47	0,61
30,0	83,86	83,01	83,49	83,42	84,96	84,48	83,77	0,73	0,87
40,0	87,11	86,41	86,40	86,82	88,22	87,89	86,89	0,77	0,88
50,0	89,43	88,64	88,98	89,13	90,67	90,12	89,05	0,78	0,88
60,0	90,75	89,77	90,46	90,74	91,88	91,19	90,46	0,72	0,79
120,0	94,70	93,89	94,02	94,66	96,42	94,97	94,23	0,94	0,99
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	87,15	88,56	86,78	87,00	85,78	88,89	85,89	1,31	1,51
15,0	110,47	110,55	109,43	110,32	110,87	112,00	109,65	0,92	0,84
20,0	122,69	122,22	122,20	122,21	123,76	123,56	122,20	0,75	0,61
30,0	134,17	132,82	133,59	133,48	135,93	135,17	134,03	1,16	0,87
40,0	139,37	138,26	138,25	138,92	141,16	140,62	139,02	1,23	0,88
50,0	143,09	141,82	142,37	142,60	145,07	144,20	142,48	1,26	0,88
60,0	145,20	143,63	144,73	145,19	147,01	145,90	144,74	1,15	0,79
120,0	151,52	150,22	150,44	151,45	154,28	151,95	150,78	1,50	0,99
0,0									
0,0									
0,0									
0,0									



AC 22.07.97



## DISSOLUTION

m:\commun\glnq\traitdon\distern5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 21/07/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2

TITRE : LF 178 TER lot C0197 LSNa 0,025M 120 tpm  
 N° CAHIER : LF 178 TER n°2 p 121  
 FICHIER : M:\commun\glnq\donnbases\LF178ter\dissolution\lot C0197 0,025M 120tpm  
 ELUANT : LSNa 0,025 M  
 AGITATION : 120 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001831

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

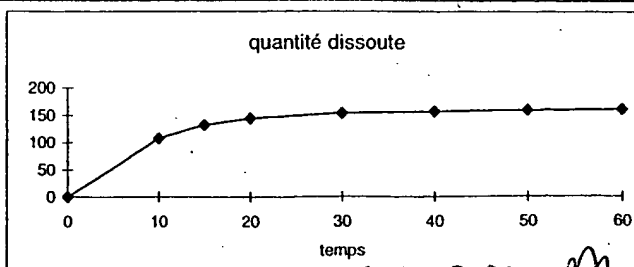
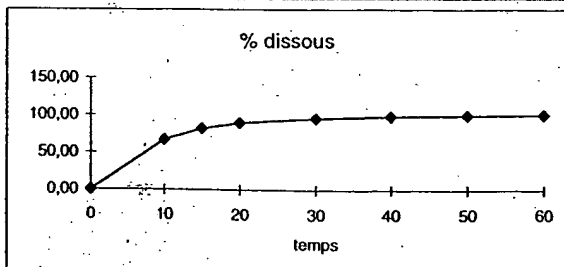
volume prélevé en ml 5		CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
TEMPS	volume en ml						
0	0	0	0	0	0	0	0
10	1000	1	1,002	0,912	0,98	1,006	0,914
15	1000	1,2	1,204	1,161	1,183	1,193	1,16
20	1000	1,297	1,293	1,278	1,277	1,291	1,272
30	1000	1,37	1,368	1,375	1,358	1,369	1,356
40	1000	1,4	1,395	1,41	1,384	1,4	1,387
50	1000	1,415	1,406	1,425	1,393	1,408	1,406
60	1000	1,419	1,408	1,431	1,401	1,411	1,412

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	67,29	69,44	69,58	63,33	68,06	69,86	63,47	3,08	4,57
15,0	82,52	83,68	83,96	80,94	82,49	83,20	80,87	1,35	1,63
20,0	89,96	90,83	90,56	89,47	89,43	90,42	89,05	0,73	0,81
30,0	96,05	96,35	96,21	96,65	95,50	96,28	95,33	0,52	0,54
40,0	98,61	98,91	98,56	99,56	97,78	98,91	97,95	0,66	0,67
50,0	99,99	100,44	99,81	101,09	98,88	99,95	99,75	0,74	0,74
60,0	100,81	101,21	100,44	102,00	99,92	100,65	100,66	0,71	0,71
0,0									
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	107,67	111,11	111,33	101,33	108,89	111,78	101,56	4,92	4,57
15,0	132,04	133,89	134,33	129,51	131,99	133,11	129,40	2,16	1,63
20,0	143,94	145,33	144,89	143,15	143,09	144,67	142,49	1,17	0,81
30,0	153,69	154,17	153,94	154,64	152,80	154,05	152,53	0,83	0,54
40,0	157,78	158,26	157,70	159,29	156,44	158,26	156,72	1,06	0,67
50,0	159,98	160,70	159,70	161,74	158,21	159,92	159,61	1,18	0,74
60,0	161,30	161,93	160,70	163,20	159,88	161,04	161,05	1,14	0,71
0,0									
0,0									
0,0									
0,0									
0,0									



PC22.07.97

12

128

21.07.97

Dissolution LF 178 ter  
lot CØ 197 - 160 mgf.  
0,05N - 50TPN

# 1. Preparation du milieu de dissolution Bal. GAZ 065

21.07.97

16:06:06

Pesée de l'eau

EAU 0.00 g

448.22 g Tare

0.00 g

3468.2 g Net

0.00 g

3916.4 g Brut

0.00 g

449.23 g Tare

-0.01 g M

0.00 g Net

3600.4 g

0.00 g

4049.6 g Brut

0.00 g

449.02 g Tare

0.00 g

3635.5 g Net

0.00 g

4084.5 g Brut

0.00 g

449.10 g Tare

0.00 g

2325.7 g Net

0.00 g

M 2774.8 g Brut

o Quantité d'eau pesée

$$3468,2 + 3600,4 + 3635,5 + 2325,7 = 13029,8 \text{ g}$$

o Quantité de LGNa à peser pour  
 un milieu à 0,05N (PM LGNa 288,4 g)

$$\frac{13029,8}{1000} \times 288,4 \times 0,05 = 187,9 \text{ g}$$

LGNa

21.07.97

16:22:31

Code

APR 1547

0.00 g

143.22 g Tare

0.00 g

187.93 g Net

0.00 g

331.15 g Brut

M

FOURNIER 1001832

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 Protective Order

RC 21.07.97

## 2. Pesée des bits de dissolution - Bal. GAL 065

129

1 litre de L90a 0.05N pèse 1002.0g.

21.07.97 16:43:13

1 0.00 g  
699.0 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1701.0 g Brut

2 0.00 g  
761.1 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1763.1 g Brut

3 0.00 g  
767.4 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1769.4 g Brut

4 0.00 g  
760.7 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1762.7 g Brut

5 0.00 g  
769.1 g Tare  
0.00 g  
1002.0 g Net  
0.00 g  
1771.1 g Brut

6 0.00 g  
753.4 g Tare  
0.01 g M  
0.01 g M  
0.00 g  
1002.0 g Net  
0.00 g  
1755.4 g Brut

## 3. Pesée des comprimés Bal. GAL 205

LF 1787ev

21.07.97 16:42:30  
Code Lot: C 0197

ID	1	2	3	4	5	6
	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
	714.3 mg	711.0 mg	719.0 mg	716.8 mg	714.4 mg	719.2 mg

## h. Conditions

Dissolutoir GAL 051

FOURNIER 1001833

Highly Confidential  
Subject to  
Protective Order

T°: 37°C ± 0.5 AC 22.07.97 M 22.07.97  
Q: 50 TPN AC 22.07.97 M 22.07.97

AC 07.97

130

S. Lechère

Spectrophotomètre GAL 283 - Chronomètre GAL 122

LF 178 TER LOT C 0197 LSNA 0.05M 50 TPM

07-22-1997 10:14

*M*

Lambda	No.	Valeur_E	
290.0	1	-0.0001_1	- Autozero Air / Air
290.0	2	0.0001_1	- LSNa / LSNa
290.0	3	0.0008_1	
290.0	4	0.0011_1	
290.0	5	0.0023_1	
290.0	6	0.0015_1	
290.0	7	0.0012_1	
290.0	8	0.0007_1	
290.0	9	0.9373_1	
290.0	10	1.1462_1	
290.0	11	0.8911_1	
290.0	12	1.0541_1	
290.0	13	1.0825_1	
290.0	14	1.0588_1	
290.0	15	1.2544_1	
290.0	16	1.3006_1	
290.0	17	1.2631_1	
290.0	18	1.3091_1	
290.0	19	1.3172_1	
290.0	20	1.3022_1	
290.0	21	1.3581_1	
290.0	22	1.3552_1	
290.0	23	1.3804_1	
290.0	24	1.3805_1	
290.0	25	1.3856_1	
290.0	26	1.3801_1	
290.0	27	1.4163_1	
290.0	28	1.3882_1	
290.0	29	1.4361_1	
290.0	30	1.4164_1	
290.0	31	1.4190_1	
290.0	32	1.4281_1	
290.0	33	1.4224_1	
290.0	34	1.3909_1	
290.0	35	1.4394_1	
290.0	36	1.4193_1	
290.0	37	1.4231_1	
290.0	38	1.4268_1	
290.0	39	1.4268_1	
290.0	40	1.3930_1	
290.0	41	1.4421_1	
290.0	42	1.4178_1	
290.0	43	1.4249_1	
290.0	44	1.4271_1	
290.0	45	1.4241_1	
290.0	46	1.3893_1	
290.0	47	1.4384_1	
290.0	48	1.4133_1	
290.0	49	1.4225_1	
290.0	50	1.4288_1	

FOURNIER 1001834

Highly Confidential  
Subject to  
Protective Order

RC 22 09 97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 22/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER lot C0197 LSNa 0,05M 50 tpm  
N° CAHIER : LF 178 TER n°2 p 128  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot C0197 0,05M 50tpm  
ELUANT : LSNa 0,05 M  
AGITATION : 50 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001835

Highly Confidential  
Subject to  
Protective Order

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

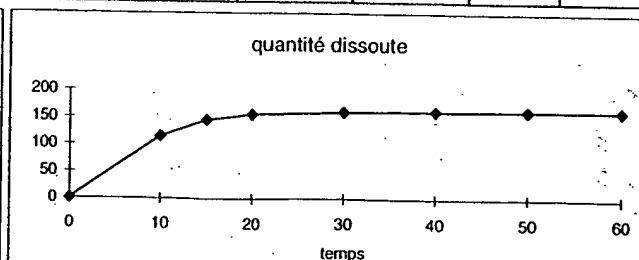
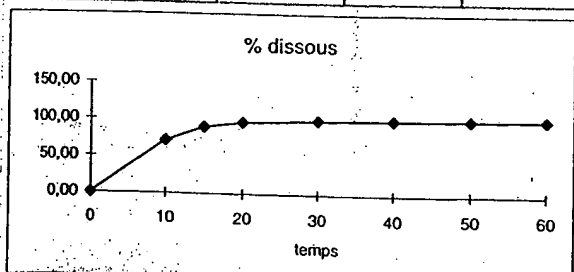
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,937	1,146	0,891	1,054	1,083	1,059
15	1000	1,254	1,301	1,263	1,309	1,317	1,302
20	1000	1,358	1,355	1,38	1,381	1,386	1,38
30	1000	1,416	1,388	1,436	1,416	1,419	1,428
40	1000	1,422	1,391	1,439	1,419	1,423	1,427
50	1000	1,427	1,393	1,442	1,418	1,425	1,427
60	1000	1,424	1,389	1,438	1,413	1,423	1,429

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	71,41	65,07	79,58	61,88	73,19	75,21	73,54	6,63	9,29
15,0	90,01	87,41	90,75	88,02	91,27	91,83	90,78	1,83	2,04
20,0	96,18	95,07	94,95	96,58	96,72	97,08	96,65	0,92	0,96
30,0	99,70	99,57	97,71	100,95	99,63	99,86	100,47	1,11	1,11
40,0	100,40	100,47	98,40	101,66	100,33	100,63	100,89	1,08	1,08
50,0	101,02	101,31	99,02	102,36	100,76	101,26	101,39	1,11	1,10
60,0	101,33	101,60	99,23	102,59	100,90	101,62	102,02	1,17	1,15
0,0									
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	114,26	104,11	127,33	99,00	117,11	120,33	117,67	10,62	9,29
15,0	144,02	139,85	145,19	140,83	146,03	146,94	145,26	2,93	2,04
20,0	153,88	152,11	151,92	154,53	154,76	155,33	154,65	1,48	0,96
30,0	159,51	159,31	156,33	161,52	159,41	159,77	160,75	1,78	1,11
40,0	160,64	160,76	157,44	162,65	160,53	161,00	161,43	1,74	1,08
50,0	161,63	162,10	158,43	163,78	161,21	162,02	162,22	1,77	1,10
60,0	162,12	162,56	158,76	164,14	161,44	162,59	163,24	1,87	1,15
0,0									
0,0									
0,0									
0,0									
0,0									



RC 22.07.97

152

22.07.97

Dissolution LF ASker  
 lot C0197 160 mg  
 0,05N - 75 TPN

# 1. Preparation du milieu de dissolution

Voir page 128

## 2. Pesée des bols de dissolution Bal. GAL 065

1 litre L<sub>3</sub>Na 0,05N pèse 1002,0 g.

22.07.97 11:10:11

1 0.00 g  
 753.4 g Tar  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1755.4 g Brut

2 0.00 g  
 767.4 g Tar  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1769.4 g Brut

3 0.00 g  
 699.3 g Tar  
 0.00 g  
 1002.1 g Net  
 0.01 g M  
 0.00 g  
 1701.4 g Brut

4 0.00 g  
 761.2 g Tar  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1763.2 g Brut

5 0.00 g  
 769.2 g Tar  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1771.2 g Brut

6 0.00 g  
 760.9 g Tar  
 -0.01 g M  
 0.00 g  
 1002.0 g Net  
 0.00 g  
 1762.9 g Brut

## 3. Pesée des comprimés Bal. GAL 205

FOURNIER 1001836  
 Highly Confidential  
 Subject to  
 Protective Order

LF ASker

22.07.97 12:36:19  
 Code Lot: C 0197

ID	1	2	3	4	5	6
	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
	714.7 mg	715.9 mg	723.2 mg	719.6 mg	715.1 mg	714.5 mg

RC 22.07.97

h. Conditions

Residual GAL 091

T° 37°C ± 0.5 AG 22/7/97 M 22.07.97

⊙ 75 TPM AG 22/7/97. M 22.07.97

LF 178 TER LOT C 0197 LSNA 0.05M 75 TPM

07-22-1997 14:12

M

S. lecture

Spectrophotometre GAL 933

Chromometre GAL 122

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0013_1
290.0	3	0.0012_1
290.0	4	0.0012_1
290.0	5	0.0010_1
290.0	6	0.0008_1
290.0	7	0.0013_1
290.0	8	0.0009_1
290.0	9	1.1794_1
290.0	10	1.1701_1
290.0	11	1.0235_1
290.0	12	1.1412_1
290.0	13	1.1603_1
290.0	14	1.1708_1
290.0	15	1.3530_1
290.0	16	1.3633_1
290.0	17	1.3223_1
290.0	18	1.3656_1
290.0	19	1.3605_1
290.0	20	1.3776_1
290.0	21	1.4095_1
290.0	22	1.4230_1
290.0	23	1.4228_1
290.0	24	1.4369_1
290.0	25	1.4236_1
290.0	26	1.4260_1
290.0	27	1.4336_1
290.0	28	1.4509_1
290.0	29	1.4603_1
290.0	30	1.4628_1
290.0	31	1.4534_1
290.0	32	1.4483_1
290.0	33	1.4336_1
290.0	34	1.4502_1
290.0	35	1.4632_1
290.0	36	1.4635_1
290.0	37	1.4525_1
290.0	38	1.4453_1
290.0	39	1.4316_1
290.0	40	1.4512_1
290.0	41	1.4631_1
290.0	42	1.4650_1
290.0	43	1.4565_1
290.0	44	1.4452_1
290.0	45	1.4214_1
290.0	46	1.4458_1
290.0	47	1.4562_1
290.0	48	1.4597_1
290.0	49	1.4488_1
290.0	50	1.4410_1

FOURNIER 1001837  
 Highly Confidential  
 Subject to  
 Protective Order

M 22.07.97

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 22/07/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2

TITRE : LF 178 TER lot C0197 LSNa 0,05M 75 tpm  
 N° CAHIER : LF 178 TER n°2 p 132  
 FICHIER : M:\commun\glnq\donbase\LF178ter\dissolution\lot C0197 0,05M 75tpm  
 ELUANT : LSNa 0,05 M  
 AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001838

Highly Confidential  
Subject to  
Protective Order

Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

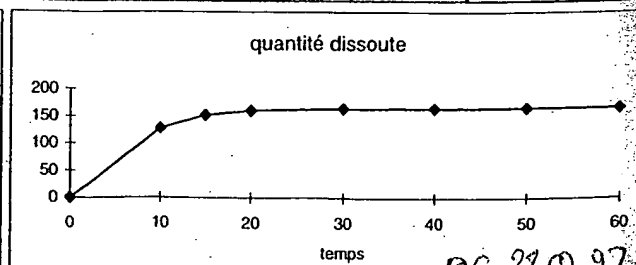
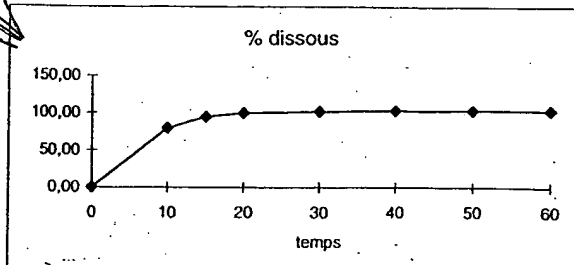
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,179	1,17	1,024	1,141	1,16	1,171
15	1000	1,353	1,363	1,322	1,366	1,361	1,378
20	1000	1,41	1,423	1,423	1,437	1,424	1,426
30	1000	1,434	1,451	1,46	1,463	1,453	1,448
40	1000	1,434	1,45	1,463	1,464	1,453	1,445
50	1000	1,432	1,451	1,463	1,465	1,457	1,445
60	1000	1,421	1,446	1,456	1,46	1,449	1,441

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	79,22	81,88	81,25	71,11	79,24	80,56	81,32	4,08	5,15
15,0	94,64	94,37	95,06	92,16	95,26	94,92	96,10	1,34	1,42
20,0	99,74	98,80	99,70	99,63	100,66	99,76	99,91	0,60	0,60
30,0	102,16	100,95	102,14	102,70	102,97	102,27	101,94	0,70	0,69
40,0	102,66	101,45	102,57	103,41	103,54	102,78	102,23	0,78	0,76
50,0	103,21	101,81	103,14	103,92	104,12	103,56	102,73	0,85	0,83
60,0	103,26	101,54	103,30	103,94	104,28	103,51	102,96	0,96	0,93
0,0									
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUTE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	126,76	131,00	130,00	113,78	126,78	128,89	130,11	6,52	5,15
15,0	151,43	150,99	152,09	147,46	152,41	151,87	153,76	2,15	1,42
20,0	159,59	158,07	159,52	159,41	161,06	159,62	159,86	0,96	0,60
30,0	163,46	161,52	163,42	164,32	164,75	163,64	163,10	1,12	0,69
40,0	164,26	162,32	164,12	165,46	165,67	164,44	163,57	1,24	0,76
50,0	165,14	162,89	165,03	166,27	166,60	165,70	164,37	1,37	0,83
60,0	165,21	162,47	165,28	166,31	166,85	165,62	164,73	1,54	0,93
0,0									
0,0									
0,0									
0,0									
0,0									



pc 2207 97



22.07.97

LF 170 Fer.

lot C0197 - 160 mg.

0,05N - 90TPN

1. Préparation du milieu de dissolution Bal. GAZ 065

EAU

22.07.97 15:42:34

0.00 g

X  
M 448.25 g Tare

0.00 g

3551.5 g Net

0.00 g

3999.7 g Brut

0.00 g

J  
M 448.68 g Tare

0.00 g

3515.9 g Net

0.00 g

3964.6 g Brut

0.00 g

Z  
M 448.93 g Tare

0.00 g

3514.9 g Net

0.00 g

3963.8 g Brut

0.00 g

449.05 g Tare

0.00 g

2510.5 g Net

0.00 g

2959.6 g Brut

M

o Quantité d'eau pesée

$$3551,5 + 3515,9 + 3514,9 + 2510,5 = 13092,8 \text{ g}$$

o Quantité de LSNa à peser pour un milieu à 0,05N

$$\frac{13092,8}{1000} \times 286,4 \times 0,05 = 188,8 \text{ g.}$$

LSNa

22.07.97 16:20:48

Code ARR 1547

0.00 g

143.29 g Tare

0.00 g

188.80 g Net

0.00 g

332.09 g Brut  
M

FOURNIER 1001839  
Highly Confidential  
Subject to  
Protective Order

PC2307.97

## 2. Remplissage des bûts de dissolution Bal. GAL 06S

1 litre de  $\text{C}_8\text{H}_{18}\text{O}_8\text{N}$  pèse 1002,0 g.

22.07.97 16:31:01

1 0.00 g  
699.0 g Tare

0.00 g

1002.0 g Net

0.00 g

1701.0 g Brut

2 0.00 g

769.0 g Tare

0.00 g

1002.0 g Net

0.00 g

1771.0 g Brut

3 -0.01 g

0.00 g

753.2 g Tare

0.00 g

1002.0 g Net

0.00 g

1755.2 g Brut

4 0.00 g

767.2 g Tare

0.00 g

1002.0 g Net

0.00 g

1769.2 g Brut

5 0.00 g

761.1 g Tare

0.00 g

1002.0 g Net

0.00 g

1763.1 g Brut

6 0.00 g

760.7 g Tare

0.00 g

1002.0 g Net

0.00 g

1762.7 g Brut

## 3. Pesée des comprimés Bal. GAL 20S

LF ABter

22.07.97 16:21:12

Code Lot: C 0197

1

0.0 mg

719.1 mg

2

0.0 mg

721.2 mg

3

0.0 mg

711.0 mg

4

0.0 mg

716.0 mg

5

0.0 mg

723.6 mg

6

0.0 mg

714.2 mg

## 4. Conditions

Dissolutest GAL 091

T° 37°C ± 0.5

Q 30 TPN

FOURNIER 1001840

Highly Confidential  
Subject to  
Protective Order

AC 23/7/97 M 23.07.97

AC 23/7/97 M 23.07.97

AC 23.07.97

S. lecture

137

spectrophotométrie GAL 183 Chromomètre GAL 182

LF 178 TER LOT C0197 LSNA 0.05M 90TPM

07-23-1997 09:03

Lambda	No.	Valeur_E
290.0	1	0.0001_1
290.0	2	0.0005_1
290.0	3	0.0014_1
290.0	4	0.0014_1
290.0	5	0.0010_1
290.0	6	0.0010_1
290.0	7	0.0020_1
290.0	8	0.0020_1
290.0	9	1.0980_1
290.0	10	1.2300_1
290.0	11	1.2452_1
290.0	12	1.2458_1
290.0	13	1.2072_1
290.0	14	1.2267_1
290.0	15	1.3508_1
290.0	16	1.3855_1
290.0	17	1.3791_1
290.0	18	1.3982_1
290.0	19	1.3752_1
290.0	20	1.3760_1
290.0	21	1.4290_1
290.0	22	1.4410_1
290.0	23	1.4245_1
290.0	24	1.4435_1
290.0	25	1.4300_1
290.0	26	1.4212_1
290.0	27	1.4602_1
290.0	28	1.4591_1
290.0	29	1.4445_1
290.0	30	1.4672_1
290.0	31	1.4553_1
290.0	32	1.4431_1

M

- Autozero Air/Air

- LSNA/LSNA

FOURNIER 1001841

Highly Confidential  
Subject to  
Protective Order

## DISSOLUTION

m:\commun\glnq\traitdon\distern5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 23/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER lot C0197 LSNa 0,05M 90 tpm  
N° CAHIER : LF 178 TER n°2 p 135 ✓  
FICHIER : M:\commun\glnq\donnbase\lf178\ter\dissolution\lot C0197 0,05M 90tpm  
ELUANT : LSNa 0,05 M ✓  
AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001842

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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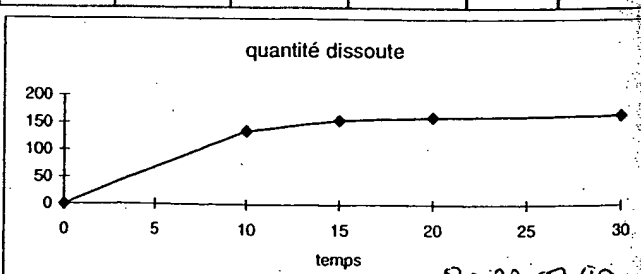
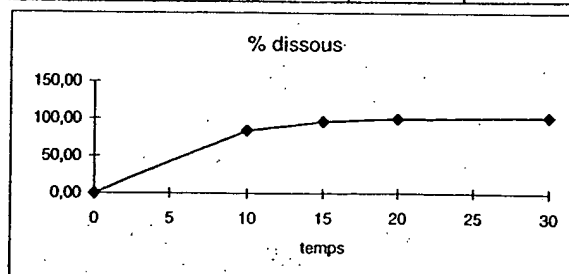
## SUIVI DE LA DISSOLUTION

[illegible]

### RÉSULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOLTE

[illegible]

RC-230792

23.07.97

Dissolution LF 178 Fe-

139

lot RG 2395/01. 14 kg.

0,05N - 50 TPN

1. Préparation du milieu de dissolution

Voir page 135

2. Remplissage des bûles de dissolution Bal. GAL 06S

Mise de LNa 0,05N pese 1002,0 g

23.07.97

11:10:51

1

0.00 g

760.9 g Taxe

0.00 g

1002.0 g Net

-0.01 g M

0.00 g

1762.9 g Brut

2

0.01 g M

0.00 g

767.4 g Taxe

0.00 g

1002.0 g Net

0.00 g

1769.4 g Brut M

3

0.00 g

769.2 g Taxe

0.00 g

1002.0 g Net

0.00 g

1771.2 g Brut

4

0.00 g

699.2 g Taxe

0.00 g

1002.0 g Net

0.00 g

1701.2 g Brut M

5

0.00 g

753.4 g Taxe

0.00 g

1002.0 g Net

0.00 g

1755.4 g Brut

6

0.00 g

761.3 g Taxe

0.00 g

1002.0 g Net

0.00 g

1763.3 g Brut M

3. Pesée des comprimés Bal. GAL 20S

FOURNIER 1001843

Highly Confidential  
Subject to  
Protective Order

LF 178 Fe

23.07.97

12:30:01

Code RG 2395/01-14 kg

1

0.0 mg

697.0 mg

2

0.0 mg

699.8 mg

3

0.0 mg

699.3 mg

4

0.0 mg

700.4 mg

5

0.0 mg

696.2 mg

6

0.0 mg

698.0 mg

RC 24.07.97 M

140 4. Conditions

Disolvent GAL 081

 $T = 37^{\circ}C \pm 0.5$  RC 23.07.97 M 23.07.97 $\Theta = 50TPM$  RC 23.07.97 M 23.07.97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.05M 50TPM

M

07-23-1997 15:07

Spectrophotometer GAL 233

Chromomètre GAL 122

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0009_1
290.0	3	0.0018_1
290.0	4	0.0014_1
290.0	5	0.0019_1
290.0	6	0.0019_1
290.0	7	0.0018_1
290.0	8	0.0022_1
290.0	9	1.0364_1
290.0	10	0.9873_1
290.0	11	1.0168_1
290.0	12	1.0460_1
290.0	13	1.0111_1
290.0	14	1.0271_1
290.0	15	1.1933_1
290.0	16	1.1577_1
290.0	17	1.2047_1
290.0	18	1.2103_1
290.0	19	1.1777_1
290.0	20	1.2065_1
290.0	21	1.2563_1
290.0	22	1.2234_1
290.0	23	1.2711_1
290.0	24	1.2797_1
290.0	25	1.2382_1
290.0	26	1.2661_1
290.0	27	1.3078_1
290.0	28	1.2842_1
290.0	29	1.3207_1
290.0	30	1.3228_1
290.0	31	1.2889_1
290.0	32	1.3174_1
290.0	33	1.3235_1
290.0	34	1.3081_1
290.0	35	1.3406_1
290.0	36	1.3403_1
290.0	37	1.3142_1

FOURNIER 1001844

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Protective Order

RC 24.07.97

## DISSOLUTION

m:\commun\glnq\traitdon\distern5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 23/07/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2

TITRE : LF 178 TER RG 2395/01 14 KG LSNa 0,05M 50tpm  
 N° CAHIER : LF 178 TER n°2 p 139  
 FICHIER : M:\commun\glnq\donnbases\LF178ter\dissolution\ot RG 2395-01 14 kg 0,05M 50tpm  
 ELUANT : LSNa 0,05 M  
 AGITATION : 50 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/l 0,900

FOURNIER 1001845

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Subject to  
Protective Order

## SUIVI DE LA DISSOLUTION

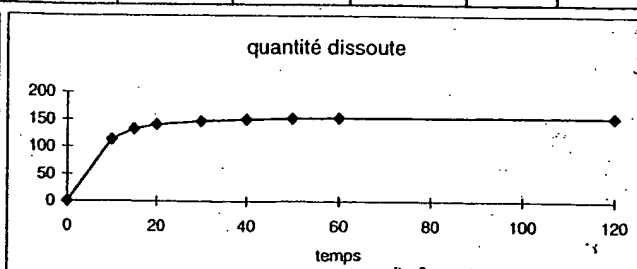
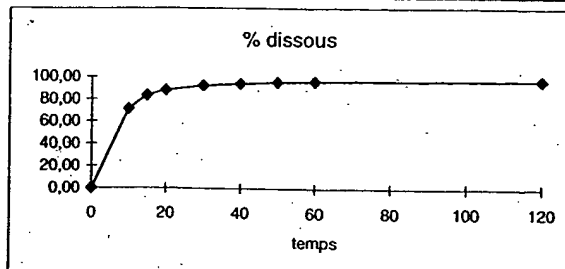
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,036	0,987	1,017	1,046	1,011	1,027
15	1000	1,193	1,158	1,205	1,21	1,178	1,207
20	1000	1,256	1,223	1,271	1,28	1,238	1,266
30	1000	1,308	1,284	1,321	1,323	1,289	1,317
40	1000	1,324	1,308	1,341	1,34	1,314	1,336
50	1000	1,335	1,324	1,357	1,356	1,323	1,344
60	1000	1,34	1,329	1,357	1,353	1,329	1,353
120	1000	1,343	1,337	1,364	1,358	1,342	1,354

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	70,88	71,94	68,54	70,63	72,64	70,21	71,32	1,44	2,04
15,0	83,12	83,21	80,76	84,03	84,39	82,16	84,18	1,42	1,71
20,0	87,97	88,00	85,68	89,04	89,67	86,73	88,69	1,51	1,71
30,0	91,97	92,04	90,34	92,95	93,10	90,70	92,67	1,18	1,29
40,0	93,82	93,61	92,45	94,80	94,74	92,89	94,45	1,00	1,07
50,0	95,16	94,83	94,01	96,37	96,32	93,97	95,47	1,07	1,13
60,0	95,88	95,64	94,82	96,84	96,58	94,84	96,56	0,91	0,95
120,0	96,78	96,32	95,84	97,80	97,40	96,21	97,10	0,77	0,79
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	113,41	115,11	109,67	113,00	116,22	112,33	114,11	2,31	2,04
15,0	132,99	133,13	129,22	134,45	135,03	131,45	134,68	2,27	1,71
20,0	140,75	140,79	137,08	142,46	143,48	138,77	141,91	2,41	1,71
30,0	147,15	147,27	144,54	148,72	148,96	145,13	148,28	1,90	1,29
40,0	150,12	149,77	147,92	151,67	151,59	148,62	151,12	1,60	1,07
50,0	152,26	151,73	150,42	154,20	154,11	150,35	152,75	1,72	1,13
60,0	153,41	153,03	151,71	154,95	154,53	151,75	154,50	1,46	0,95
120,0	154,84	154,11	153,34	156,48	155,84	153,93	155,36	1,23	0,79
0,0									
0,0									
0,0									
0,0									



PC 24.07.97

142

23.07.97

Gelules Lipidil Micro Leolot h<sup>9</sup> CanadaLSNa 0,1 N - 30 TPN1. Préparation du milieu de dissolution Bal. GAL 070

23.07.97

15:59:03

EAU 0.00 g

448.26 g Tare

~~-0.01 g M~~

0.00 g

3544.9 g Net

~~0.00 g~~

3993.2 g Brut

0.00 g

448.73 g Tare

0.00 g

3547.1 g Net

0.00 g

3995.8 g Brut

0.00 g

448.99 g Tare

0.00 g

3468.7 g Net

0.00 g

3917.7 g Brut

0.00 g

449.22 g Tare

0.00 g

2531.5 g Net

0.00 g

M 2980.7 g Brut

o Quantité d'eau pesée

$$3544.9 + 3547.1 + 3468.7 + 2531.5 = 13092.2 \text{ g}$$

o Quantité de LSNa à peser pour un milieu à 0,1 N

$$\frac{13092.2}{1000} \times 288.4 \times 0.1 = 377.6 \text{ g}$$

LSNa

23.07.97

16:25:19

Code

ARN

1547

0.00 g

448.24 g Tare

0.00 g

377.60 g Net

0.00 g

825.8 g Brut

M

FOURNIER 1001846

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Protective Order

RC 24.07.97



## 2. Remplissage des bds de dissolution Bal. GAL 070

143

Alite de L'Na 0.11 pèse 1004.0 g.

23.07.97 16:50:02

1 0.00 g  
761.00 g Tare  
0.00 g  
1004.0 g Net  
0.00 g  
1765.0 g Brut

2 0.00 g

23.07.97 16:52:52

M 698.97 g M  
0.00 g Tare  
1004.0 g Net  
0.00 g

M 1703.0 g Brut

3 0.00 g  
767.18 g Tare  
0.00 g  
1004.0 g Net  
0.00 g  
1771.2 g Brut

4 0.00 g

760.69 g Tare

0.00 g  
1004.0 g Net  
0.00 g

M 1764.7 g Brut

5 0.00 g  
753.18 g Tare  
0.00 g  
1004.0 g Net  
0.00 g  
1757.2 g Brut

6 -0.01 g M

0.00 g

769.00 g Tare  
0.00 g  
1004.0 g Net  
0.00 g

M 1773.0 g Brut

M

M

## 3. Pesée des gélules Bal. GAL 205

Lipidel Nivo 200

23.07.97 16:40:30

Code Canada lot 49

ID	1	2	3	4	5	6
0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
428.8 mg	428.8 mg	431.0 mg	428.3 mg	-0.1 mg	429.2 mg	436.4 mg
423.6 mg						

## h. Conditions

Dissolubest GAL 091

$T = 37^{\circ}\text{C} \pm 0.5$  Re 24.07.97 M 24.07.97

$\phi = 90$  TPN Re 24.07.97 M 24.07.97

FOURNIER 1001847

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Subject to  
Protective Order

RC 24.07.97

S. lecher

Spectrophotomètre GAL 233  
 Chronomètre GAL 122

GELULES LIPIDIL MICRO LOT 49 CANADA LSNA 0.1M 90TPM

07-24-1997 10:30

Lambda No. Valeur\_E

M

290.0	1	-0.0000_1
290.0	2	0.0002_1
290.0	3	0.0016_1
290.0	4	0.0007_1
290.0	5	0.0013_1
290.0	6	0.0016_1
290.0	7	0.0024_1
290.0	8	0.0008_1
290.0	9	0.5693_1
290.0	10	0.5839_1
290.0	11	0.3920_1
290.0	12	0.7213_1
290.0	13	0.2582_1
290.0	14	0.3198_1
290.0	15	1.1405_1
290.0	16	1.2041_1
290.0	17	0.9408_1
290.0	18	1.2498_1
290.0	19	0.7509_1
290.0	20	0.9271_1
290.0	21	1.4010_1
290.0	22	1.4447_1
290.0	23	1.3140_1
290.0	24	1.4429_1
290.0	25	1.2594_1
290.0	26	1.3121_1
290.0	27	1.5707_1
290.0	28	1.6046_1
290.0	29	1.5400_1
290.0	30	1.5875_1

FOURNIER 1001848

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 Protective Order

08/18/97

# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

145

## CONDITIONS ET MODES OPERATOIRES

OPÉRATEUR : D.LECRIT  
DATE : 24/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : Gelules Lipidil Micro lot 49 Canada LSNa 0,1M 90 tpm  
N° CAHIER : LF 178 TER n°2 p 142  
FICHIER : M:\commun\glnq\donnbases\LF178ter\dissolution\lot 49 Canada 0,1M 90tpm  
ELUANT : LSNa 0,1 M  
AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 350  
dosage théorique 200 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	350,00	350,00	350,00	350,00	350,00	350,00
quantité de principe actif	200,00	200,00	200,00	200,00	200,00	200,00

Témoin 100mg/l 0,900

FOURNIER 1001849

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## SUIVI DE LA DISSOLUTION

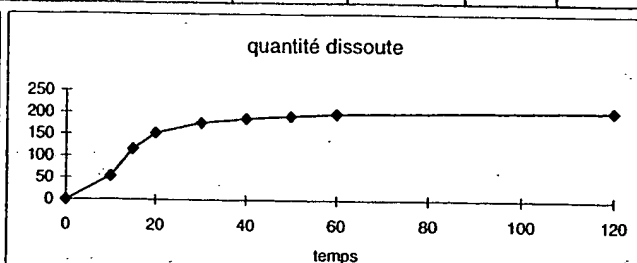
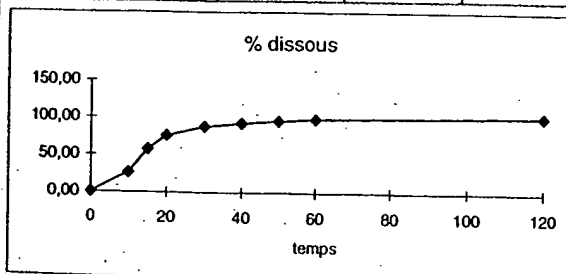
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,569	0,584	0,392	0,721	0,258	0,32
15	1000	1,141	1,204	0,941	1,25	0,751	0,927
20	1000	1,401	1,445	1,314	1,443	1,259	1,312
30	1000	1,571	1,605	1,54	1,588	1,56	1,551
40	1000	1,646	1,674	1,627	1,656	1,652	1,631
50	1000	1,694	1,72	1,686	1,699	1,708	1,681
60	1000	1,724	1,754	1,727	1,728	1,747	1,716
120	1000	1,794	1,802	1,795	1,787	1,83	1,784

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	26,33	31,61	32,44	21,78	40,06	14,33	17,78	9,91	37,65
15,0	57,67	63,55	67,05	52,39	69,64	41,79	51,59	10,80	18,72
20,0	76,10	78,31	80,77	73,37	80,71	70,22	73,24	4,43	5,82
30,0	87,97	88,14	90,06	86,29	89,17	87,30	86,88	1,44	1,63
40,0	92,77	92,75	94,34	91,55	93,39	92,84	91,75	1,04	1,12
50,0	96,02	95,87	97,36	95,28	96,24	96,41	94,98	0,85	0,89
60,0	98,42	98,01	99,73	98,03	98,32	99,05	97,40	0,84	0,85
120,0	102,57	102,37	102,89	102,29	102,08	104,15	101,65	0,87	0,85
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	52,67	63,22	64,89	43,56	80,11	28,67	35,56	19,83	37,65
15,0	115,34	127,09	134,10	104,77	139,29	83,59	103,18	21,59	18,72
20,0	152,21	156,62	161,55	146,74	161,43	140,45	146,47	8,86	5,82
30,0	175,95	176,28	180,13	172,58	178,34	174,59	173,76	2,87	1,63
40,0	185,54	185,49	188,69	183,10	186,78	185,68	183,51	2,08	1,12
50,0	192,05	191,74	194,73	190,56	192,48	192,82	189,97	1,71	0,89
60,0	196,84	196,01	199,46	196,06	196,64	198,10	194,79	1,67	0,85
120,0	205,14	204,75	205,77	204,57	204,16	208,30	203,30	1,74	0,85
0,0									
0,0									
0,0									
0,0									



SC 08/08/97

146  
24.07.97

Dissolution LF 178ter  
lot C0187 - 160 mg  
0,1M - 50 TPN

1. Préparation du milieu de dissolution Bal GAL 665

Voir page 142

2. Remplissage des bds de dissolutions Bal GAL 665  
1 litre LSN 0,1M pte 100h, 0g

24.07.97 11:18:55

1 0.00 g  
753.4 g Taxe  
0.00 g

1004.0 g Net

0.00 g

1757.4 g Brut

2 0.00 g  
760.9 g Taxe  
0.00 g

1004.0 g Net

0.00 g

1764.9 g Brut

3 0.00 g  
699.1 g Taxe  
0.01 g M  
0.00 g

1004.0 g Net

0.00 g

1703.1 g Brut

4 0.00 g  
767.4 g Taxe  
0.00 g

1004.0 g Net

0.00 g

1771.3 g Brut

5 0.00 g  
761.1 g Taxe  
-0.01 g M  
0.00 g

1004.0 g Net

0.00 g

1765.1 g Brut

6 0.00 g  
769.3 g Taxe  
0.00 g

1004.0 g Net

0.00 g

1773.3 g Brut

3. Pesée des comprimés Bal GAL 205

LF 178ter  
24.07.97 10:09:58  
Code lot: C 0197

ID 1

0.0 mg

709.5 mg

ID 2

0.0 mg

716.3 mg

ID 3

0.0 mg

721.7 mg

ID 4

719.9 mg

0.0 mg

719.9 mg

ID 5

0.0 mg

711.6 mg

ID 6

0.0 mg

719.9 mg

FOURNIER 1001850  
Highly Confidential  
Subject to  
Protective Order

08/08/97

# h. Conditions

147

Dissealtest GAL 001

T° = 37°C ± 0.5 RC 24.07.97 M 24.07.97

Q = 50 TPM RC 24.07.97 M 24.07.97

## 5. Lecture

Spectrophotomètre GAL 233

Chronomètre GAL 122

LF 178 TER LOT C0197 LSNA 0.1 M 50 TPM

07-24-1997 13:53

Lambda No. Valeur\_E

290.0	1	0.0000_1
290.0	2	0.0005_1
290.0	3	0.0009_1
290.0	4	0.0008_1
290.0	5	0.0012_1
290.0	6	0.0009_1
290.0	7	0.0025_1
290.0	8	0.0013_1
290.0	9	1.2092_1
290.0	10	0.9001_1
290.0	11	0.9888_1
290.0	12	1.0187_1
290.0	13	1.1309_1
290.0	14	1.2426_1
290.0	15	1.3693_1
290.0	16	1.3207_1
290.0	17	1.3706_1
290.0	18	1.3643_1
290.0	19	1.3762_1
290.0	20	1.3834_1
290.0	21	1.4000_1
290.0	22	1.3948_1
290.0	23	1.4295_1
290.0	24	1.4283_1
290.0	25	1.4138_1
290.0	26	1.4074_1
290.0	27	1.4171_1
290.0	28	1.4299_1
290.0	29	1.4596_1
290.0	30	1.4596_1
290.0	31	1.4339_1
290.0	32	1.4201_1

M  
Autogero Air/Air  
LSNa / LSNa

FOURNIER 1001851

Highly Confidential  
Subject to  
Protective Order

SL 08/08/92

## DISSOLUTION

m:\commun\ginq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 24/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2

TITRE : LF 178 TER lot C0197 LSNa 0,1M 50 tpm  
N° CAHIER : LF 178 TER n°2 p 146  
FICHER : M:\commun\ing\donnbase\LF178ter\dissolution\lot C0197 0,1M 50tpm  
ELUANT : LSNa 0,1 M  
AGITATION : 50 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

**FOURNIER 1001852**

**Highly Confidential  
Subject to  
Protective Order.**

Témoin 100mg/l	0,900
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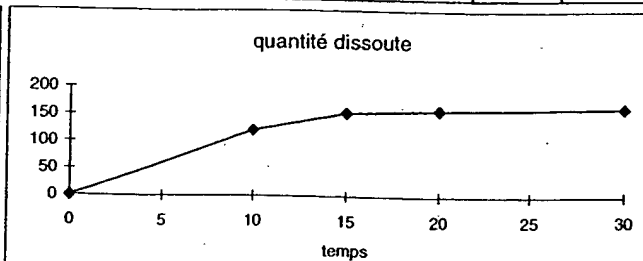
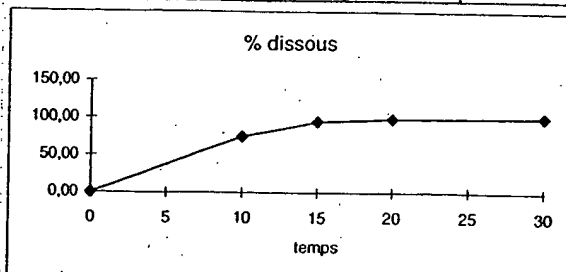
### SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOUTE

[illegible]

24.07.97

Dissolution LS ARR ter

149

ldr CQ187 - 160mg.0.1N - 75 TPN

1. Préparation du milieu de dissolution Bal. GAT 06  
 Pesée de l'eau :

EAU  
 24.07.97 15:30:16  
 0.00 g

448.28 g Tare

0.00 g

3498.1 g Brut

0.00 g

3946.4 g Net

0.00 g

449.18 g Tare

0.00 g

3505.3 g Brut

0.00 g

3954.5 g Brut

o Quantité d'eau pesée

$$3498.1 + 3505.3 = 7003.4 \text{ g}$$

o Quantité de LSNa à peser  
 pour un milieu à 0.1N

$$\frac{7003.4}{1000} \times 286.4 \times 0.1 = 202.0 \text{ g}$$

LSNa ARR 1547

24.07.97 15:43:29

0.00 g

143.23 g Tare

0.00 g

202.02 g Net

~~143.23 g~~ M

0.00 g

345.25 g Brut

M

FOURNIER 1001853

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FL 08/02/97

150

## 2. Remplissage des bts de dissolution Bal. GAL 065

Mise de 18Na 0,1M. pèse 100h, 0 p.

24.07.97 15:57:25

1 0.00 g  
699.1 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1703.1 g Brct

2 0.00 g  
761.0 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1765.0 g Brct M

3 0.00 g  
753.2 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1757.2 g Brct

4 0.00 g  
760.7 g Tax  
0.00 g  
1004.0 g Net  
-0.01 g M  
0.00 g  
1764.7 g Brct M

5 0.00 g  
767.2 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1771.2 g Brct

6 0.00 g  
769.0 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1773.0 g Brct M

## 3. Pesée des compaires Bal. GAL 005

LF 17064  
24.07.97 15:30:25  
Code 100 C 0197

ID	1	2	3	4	5	6
	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
	711.2 mg	717.0 mg	707.0 mg	714.0 mg	715.2 mg	717.0 mg

## 4. Conditions

Dissolution GAL 001

T° 37°C ± 0.5 RC 25.07.97 M 25.07.97

Θ 75 TPN RC 25.07.97 M 25.07.97

08/08/97

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5. Lecture

Spectrophotométre GAL 233  
Chromométre GAL 122

LF 178 TER LOT C0197 LSNA 0.1M 75TPM

M 07-25-1997 09:24

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0014_1
290.0	3	0.0025_1
290.0	4	0.0020_1
290.0	5	0.0011_1
290.0	6	0.0017_1
290.0	7	0.0013_1
290.0	8	0.0012_1
290.0	9	1.1840_1
290.0	10	1.0710_1
290.0	11	1.1889_1
290.0	12	1.2799_1
290.0	13	1.3011_1
290.0	14	1.0664_1
290.0	15	1.3948_1
290.0	16	1.3924_1
290.0	17	1.3965_1
290.0	18	1.4245_1
290.0	19	1.4142_1
290.0	20	1.3894_1
290.0	21	1.4320_1
290.0	22	1.4466_1
290.0	23	1.4282_1
290.0	24	1.4490_1
290.0	25	1.4384_1
290.0	26	1.4456_1
290.0	27	1.4400_1
290.0	28	1.4538_1
290.0	29	1.4365_1
290.0	30	1.4502_1
290.0	31	1.4436_1
290.0	32	1.4543_1

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92-08/08/88

## DISSOLUTION

m:\commun\qing\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 25/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2

TITRE : LF 178 TER lot C0197 LSNa 0,1M 75 tpm  
N° CAHIER : LF 178 TER n°2 p 149  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot C0197 0,1M 75tpm  
ELUANT : LSNa 0,1 M  
AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

masse de la prise d'essai  
quantité de principe actif

CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
160,00	160,00	160,00	160,00	160,00	160,00
160,00	160,00	160,00	160,00	160,00	160,00

FOURNIER 1001856

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Témoin 100mg/l	0.900
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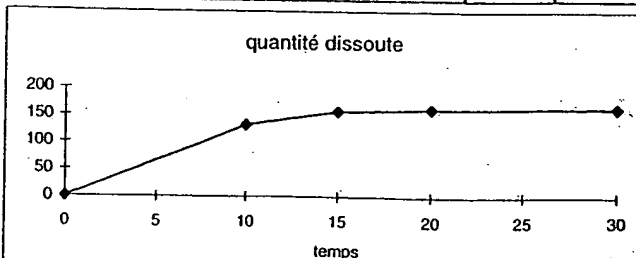
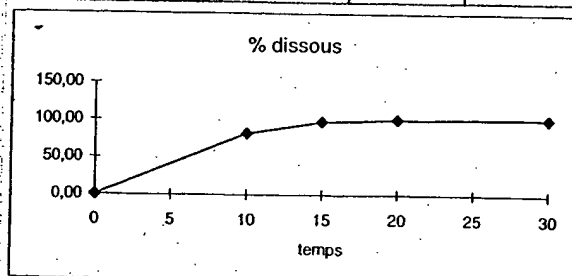
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOUTE

[illegible]

28.07.97

Dissolution LF 178 ter

lot C0137 - 160 mg.

0,1N - 120 TPN

152'

Encre à page  
fin voir page  
page 10

1. Pesée de l'eau M

1. Préparation du milieu de dissolution Bal GAL 065

28.07.97

07:54:28

Pesée de l'eau

EAU

0.00 g

448.23 g Tare

0.00 g

3425.0 g Net

0.00 g

3873.2 g Brut

0.00 g

448.46 g Tare

0.00 g

3489.3 g Net

0.00 g

3937.7 g Brut

0.00 g

448.52 g Tare

-0.01 g M

0.00 g

3513.6 g Net

0.00 g

3962.1 g Brut

0.00 g

448.47 g Tare

0.00 g

2462.7 g Net

0.00 g

2911.2 g Brut

M

o Quantité d'eau pesée:

$$3425.0 + 3489.3 + 3513.6 + 2462.7 = 12890.6g$$

o Pesée du L8Ne

Pour un milieu à 0,1N →

$$\frac{12890.6}{1000} \times 288.1 \times 0.1 = 371.8 g.$$

L8Ne

28.07.97

08:22:01

Code

ARR

1547

0.00 g

448.24 g Tare

0.00 g

371.80 g Net

0.00 g

820.0 g Brut

M

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28/08/97

## 2. Remplissage des bts de dissolution, Bal. GAL 065

Uché LSNa 0.1M pese 100h, 0g.

28.07.97 08:48:06

1 0.00 g  
753.0 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1757.0 g Brut

2 0.00 g  
767.0 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1771.0 g Brut  
M

3 0.00 g  
698.8 g Tax  
0.00 g  
1004.0 g Net  
0.01 g M  
0.00 g  
1702.8 g Brut

4 0.00 g  
760.5 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1764.5 g Brut  
M

5 0.00 g  
768.8 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1772.8 g Brut

6 0.00 g  
760.8 g Tax  
0.00 g  
1004.0 g Net  
0.00 g  
1764.8 g Brut

## 3. Pesée des comprimés Bal. GAL 205.

LF 17070

28.07.97 09:34:01  
Code 10197

ID 1

0.0 mg

719.5 mg

ID 2

0.0 mg

722.1 mg

ID 3

0.0 mg

713.8 mg

ID 4

0.0 mg

719.2 mg

ID 5

0.0 mg

715.5 mg

ID 6

0.0 mg

715.4 mg

## h. Conditions

Dissolvant GAL 091

T° = 37°C ± 0.5

Q = 120 TPN

28/07/97 M 26.07.97

28/07/97 M 26.07.97

28/08/97

FOURNIER 1001858

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Subject to  
Protective Order

5. Lecture

Spectrophotomètre GAL 833

Chronomètre GAL 122

LF 178 TER LOT C0197 LSNA 0.1M 120 TPM

M 07-28-1997 10:33

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0002_1
290.0	3	0.0021_1
290.0	4	0.0006_1
290.0	5	0.0028_1
290.0	6	0.0006_1
290.0	7	0.0007_1
290.0	8	0.0027_1
290.0	9	1.3303_1
290.0	10	1.3497_1
290.0	11	1.3419_1
290.0	12	1.2525_1
290.0	13	1.3471_1
290.0	14	1.3533_1
290.0	15	1.4347_1
290.0	16	1.4337_1
290.0	17	1.4329_1
290.0	18	1.4317_1
290.0	19	1.4283_1
290.0	20	1.4348_1
290.0	21	1.4427_1
290.0	22	1.4515_1
290.0	23	1.4484_1
290.0	24	1.4550_1
290.0	25	1.4442_1
290.0	26	1.4491_1

FOURNIER 1001859

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Subject to  
Protective Order

## DISSOLUTION

m:\commun\qlng\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 28/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2

TITRE : LF 178 TER lot C0197 LSNa 0,1M 120 tpm  
N° CAHIER : LF 178 TER n°2 p 152  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot C0197 0,1M 120tpm  
ELUANT : LSNa 0,1 M  
AGITATION : 120 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

**FOURNIER 1001860**

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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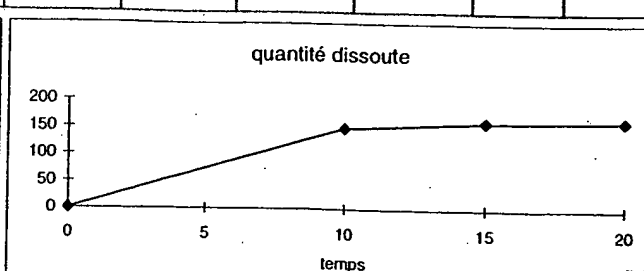
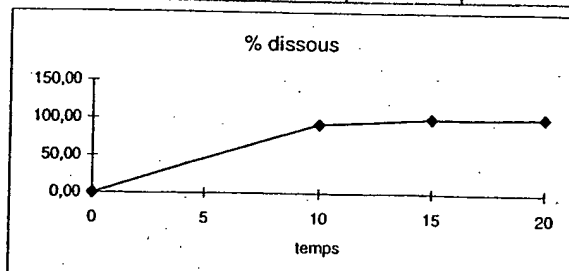
### SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOUTE

[illegible]

522 08/08/98

26.07.97

Dissolution - LF-178100

151

Ltr RG 2395/01 14 kg.

0.1N - 50 TPN

## 1. Réparation du milieu de dissolution

Voir page 152

## 2. Pesée des bds de dissolution Bal GALCOS

28.07.97 11:06:41

1 0.00 g  
699.1 g Taxe  
0.00 g  
1004.0 g Net  
0.00 g  
1703.1 g Brut

2 0.00 g  
769.1 g Taxe  
0.00 g  
1004.0 g Net  
0.00 g  
1773.1 g Brut m

3 0.00 g  
753.3 g Taxe  
0.00 g  
1004.0 g Net  
0.00 g  
1757.3 g Brut

4 0.00 g  
761.1 g Taxe  
0.00 g  
1004.0 g Net  
0.00 g  
1765.1 g Brut m

5 0.00 g  
760.8 g Taxe  
0.00 g  
1004.0 g Net  
1764.8 g Brut

6 0.00 g  
767.3 g Taxe  
0.00 g  
1004.0 g Net  
0.00 g  
1771.3 g Brut m

publi  
d'impress  
de la  
remise  
zéro.

26.07.97

Plute de 1.8Na 0.1N pese 100h Op.

## 3. Pesée des comprimés Bal GALCOS

FOURNIER 1001861

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Subject to  
Protective Order

LF-178100  
28.07.97 12:37:06  
Code RG 2395/01-14kg  
ID 1 0.0 mg  
2 699.3 mg  
3 0.0 mg  
4 703.5 mg  
5 0.0 mg  
6 704.4 mg  
7 0.0 mg  
8 703.4 mg  
9 0.0 mg  
10 702.9 mg  
11 0.0 mg  
12 705.0 mg

2 02/08/97

157

h. Conditions

R8804441 BAL 091

T° 37°C ± 0,5 SC 28/07/97 M 28.07.97

G 30 TPN 2 28/07/97 M 28.07.97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.1M 50 TPM

Lambda	No.	Valeur_E
290.0	1	-0.0000_1
290.0	2	0.0012_1
290.0	3	0.0012_1
290.0	4	0.0017_1
290.0	5	0.0017_1
290.0	6	0.0007_1
290.0	7	0.0011_1
290.0	8	0.0013_1
290.0	9	1.0798_1
290.0	10	1.0940_1
290.0	11	1.1154_1
290.0	12	1.1175_1
290.0	13	1.0779_1
290.0	14	1.0668_1
290.0	15	1.2286_1
290.0	16	1.2463_1
290.0	17	1.2464_1
290.0	18	1.2525_1
290.0	19	1.2355_1
290.0	20	1.2414_1
290.0	21	1.2800_1
290.0	22	1.3014_1
290.0	23	1.2910_1
290.0	24	1.3071_1
290.0	25	1.2909_1
290.0	26	1.3031_1
290.0	27	1.3184_1
290.0	28	1.3402_1
290.0	29	1.3217_1
290.0	30	1.3361_1
290.0	31	1.3262_1
290.0	32	1.3481_1
290.0	33	1.3320_1
290.0	34	1.3550_1
290.0	35	1.3369_1
290.0	36	1.3455_1
290.0	37	1.3475_1
290.0	38	1.3665_1
290.0	39	1.3368_1
290.0	40	1.3629_1
290.0	41	1.3427_1
290.0	42	1.3513_1
290.0	43	1.3493_1
290.0	44	1.3740_1
290.0	45	1.3347_1
290.0	46	1.3630_1
290.0	47	1.3409_1
290.0	48	1.3516_1
290.0	49	1.3537_1
290.0	50	1.3732_1

07-28-1997 13:56

FOURNIER 1001862

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Subject to  
Protective Order

2 28/08/97



# DISSOLUTION

m:\commun\glh\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 28/07/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm : 2

TITRE : LF 178 TER lot RG 2395/01 14 kg LSNa 0,1M 50 tpm  
 N° CAHIER : LF 178 TER n°2 p 156  
 FICHIER : M:\commun\glh\donnbases\LF178ter\dissolution\lot RG 2395-01 14 kg 0,1M 50tpm  
 ELUANT : LSNa 0,1 M  
 AGITATION : 50 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
 dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

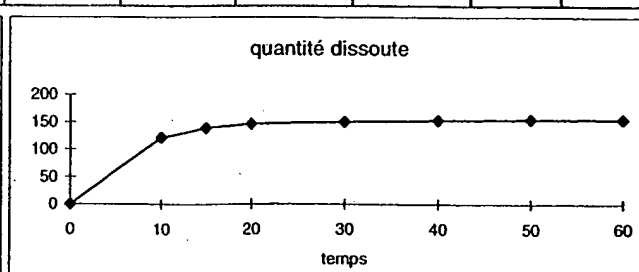
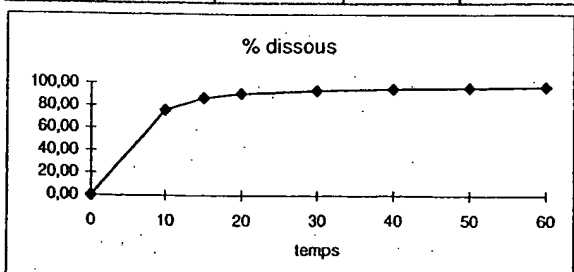
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,08	1,094	1,115	1,118	1,078	1,067
15	1000	1,229	1,246	1,246	1,253	1,236	1,241
20	1000	1,28	1,301	1,291	1,307	1,291	1,303
30	1000	1,318	1,34	1,322	1,336	1,326	1,348
40	1000	1,332	1,355	1,337	1,346	1,348	1,367
50	1000	1,337	1,363	1,343	1,351	1,349	1,374
60	1000	1,335	1,363	1,341	1,352	1,354	1,373

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	75,83	75,00	75,97	77,43	77,64	74,86	74,10	1,45	1,91
15,0	86,62	85,72	86,91	86,91	87,40	86,21	86,55	0,59	0,69
20,0	90,78	89,69	91,16	90,47	91,59	90,46	91,29	0,70	0,77
30,0	93,74	92,77	94,32	93,07	94,05	93,34	94,86	0,81	0,86
40,0	95,30	94,20	95,83	94,57	95,21	95,32	96,65	0,88	0,92
50,0	96,14	95,01	96,85	95,46	96,03	95,86	97,61	0,95	0,99
60,0	96,62	95,34	97,33	95,78	96,57	96,68	98,02	0,98	1,02
0,0									
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	121,33	120,00	121,56	123,89	124,22	119,78	118,56	2,32	1,91
15,0	138,59	137,16	139,05	139,06	139,84	137,93	138,48	0,95	0,69
20,0	145,24	143,51	145,86	144,76	146,54	144,73	146,06	1,12	0,77
30,0	149,98	148,44	150,91	148,92	150,49	149,34	151,78	1,29	0,86
40,0	152,48	150,73	153,32	151,32	152,34	152,52	154,64	1,40	0,92
50,0	153,82	152,02	154,96	152,73	153,64	153,38	156,18	1,52	0,99
60,0	154,59	152,54	155,72	153,25	154,51	154,68	156,83	1,57	1,02
0,0									
0,0									
0,0									
0,0									
0,0									



52 00/08/97

159

28.07.97

F178 ker. Dissolution

RG 2385/01. 14 kg.

LSNa 0,05N - 75 TPN

# 1. Preparation du milieu de dissolution Bal. GAL 065

Pesée de l'eau

EAU

28.07.97 14:43:19

0.00 g

448.24 g Tare

0.00 g

3487.1 g Net -

0.00 g

3935.4 g Brct

0.00 g

448.52 g Tare

0.00 g

3513.3 g Net -

0.00 g

3961.8 g Brct

0.00 g

448.49 g Tare

0.00 g

3518.3 g Net -

0.00 g

3966.7 g Brct

0.00 g

448.47 g Tare

0.00 g

2462.9 g Net -

0.00 g

2911.3 g Brct

M

quantité d'eau pesée

$$3487,1 + 3513,3 + 3518,3 + 2462,9 = 12981,6 \text{ g}$$

quantité de LSNa à peser pour un milieu à 0,05N

$$\frac{12981,6}{1000} \times 288,4 \times 0,05 = 187,2 \text{ g}$$

LSNa

28.07.97

15:02:44

Code

APR 1547

0.00 g

448.24 g Tare

0.01 g M

0.00 g

187.21 g Net -

0.00 g

635.5 g Brct

M

FOURNIER 1001864

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08/08/97

## 2. Remplissage des bols de dissolution. Bal. GAL 065 160

Alitre de 1800a 0.05N pèse 1002.0g

28.07.97 15:39:53

1 0.00 g  
753.2 g Take  
0.00 g  
1002.0 g Net -  
0.00 g  
1755.2 g Brck

2 0.00 g  
760.7 g Take  
0.00 g  
1002.0 g Net -  
0.00 g  
1762.7 g Brck M

3 0.00 g  
769.0 g Take  
0.00 g  
1002.0 g Net -  
0.00 g  
1771.0 g Brck

4 0.00 g  
699.0 g Take  
0.00 g  
1002.0 g Net -  
0.00 g  
1701.0 g Brck M

5 0.00 g  
767.1 g Take  
0.00 g  
1002.0 g Net -  
0.00 g  
1769.1 g Brck

6 0.01 g M  
0.00 g  
761.0 g Take  
0.00 g  
1002.0 g Net -  
0.00 g  
1763.0 g Brck M

## 3. Pesée des comprimés Bal. GAL 80S

15 Absolu  
28.07.97 16:02:35  
Code 26 2395/01-146g

ID	1	2	3	4	5	6
	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg	0.0 mg
	703.0 mg	690.1 mg	704.2 mg	701.7 mg	701.9 mg	702.1 mg

## h. Conditions

Dissolutest GAL 081

T° 37° ± 0.5 SC 28/07/97 M 28.07.97  
Q 75 TPN Q 28/08/97 M 28.07.97

FOURNIER 1001865  
Highly Confidential  
Subject to  
Protective Order

SC 08/08/97

5. lectureSpectrophotomètre GAL 233  
Chronomètre GAL 122

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.05M 75 TPM

M

07-29-1997 09:20

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0009_1
290.0	3	0.0017_1
290.0	4	0.0013_1
290.0	5	0.0020_1
290.0	6	0.0017_1
290.0	7	0.0017_1
290.0	8	0.0014_1
290.0	9	1.1607_1
290.0	10	1.1760_1
290.0	11	1.1981_1
290.0	12	1.1691_1
290.0	13	1.1416_1
290.0	14	1.1710_1
290.0	15	1.2800_1
290.0	16	1.2787_1
290.0	17	1.3048_1
290.0	18	1.2834_1
290.0	19	1.2692_1
290.0	20	1.2903_1
290.0	21	1.3201_1
290.0	22	1.3194_1
290.0	23	1.3474_1
290.0	24	1.3314_1
290.0	25	1.3130_1
290.0	26	1.3262_1
290.0	27	1.3436_1
290.0	28	1.3393_1
290.0	29	1.3691_1
290.0	30	1.3552_1
290.0	31	1.3440_1
290.0	32	1.3490_1
290.0	33	1.3487_1
290.0	34	1.3380_1
290.0	35	1.3766_1
290.0	36	1.3554_1
290.0	37	1.3489_1
290.0	38	1.3566_1
290.0	39	1.3537_1
290.0	40	1.3416_1
290.0	41	1.3772_1
290.0	42	1.3606_1
290.0	43	1.3548_1
290.0	44	1.3609_1
290.0	45	1.3511_1
290.0	46	1.3393_1
290.0	47	1.3788_1
290.0	48	1.3656_1
290.0	49	1.3591_1
290.0	50	1.3599_1

FOURNIER 1001866

Highly Confidential  
Subject to  
Protective Order

32 08/08/90

## DISSOLUTION

m:\commun\glnq\traitdon\distern5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 29/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm: 2

TITRE : LF 178 TER lot RG 2395/01 14 kg LSNa 0,05M 75 tpm  
N° CAHIER : LF 178 TER n°2 p 159  
FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot RG 2395-01 14 kg 0,05M 75tpm  
ELUANT : LSNa 0,05M  
AGITATION : 75 TPM

### PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/l	0,900
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FOURNIER 1001867

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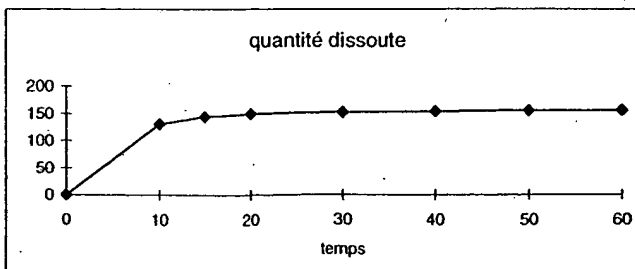
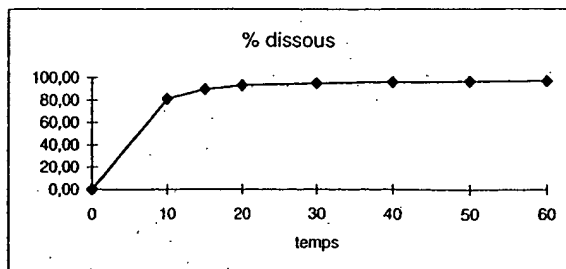
## SUIVI DE LA DISSOLUTION

[illegible]

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOLTE

[illegible]

5/08/2022

163

29.07.97

Dissolution LF 176 TerLot RG 2385/01 1 kgLSNa 0,05N - 90 TPN1. Préparation du milieu de dissolution

Voir page 159

2. Remplissage des bds de dissolution Bal. GAZ 065  
1 litre de LSNa 0,05N pèse 1002,0 g.

29.07.97 10:52:10

1 0.00 g

767.5 g Tax

0.00 g

1002.0 g Net

0.00 g

1769.5 g Brut

3 0.00 g

760.9 g Tax

0.00 g

1002.0 g Net

0.00 g

1762.9 g Brut

5 0.00 g

761.2 g Tax

0.00 g

1002.0 g Net

0.00 g

1763.2 g Brut

2 0.00 g

769.2 g Tax

0.00 g

1002.0 g Net

0.00 g

1771.2 g Brut

4 0.00 g

753.4 g Tax

0.00 g

1002.0 g Net

0.00 g

1755.4 g Brut

6 0.00 g

699.2 g Tax

0.00 g

1002.0 g Net

0.00 g

1701.2 g Brut

3. Pesée des comprimés Bal. GAZ 205

FOURNIER 1001868

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29.07.97 11:13:05

Code RG 2385/01-14 kg

ID 1

0.0 mg

700.7 mg

ID 2

0.0 mg

703.3 mg

ID 3

0.0 mg

702.0 mg

ID 4

0.0 mg

704.0 mg

ID 5

0.0 mg

704.4 mg

ID 6

0.0 mg

707.9 mg

08/08/97

## h. Conditions

Dissolved GAL 031

T° = 37°C ± 0.5 &amp; 28/07/97 M 29.07.97

Q = 90 TPR &amp; 28/07/97 M 29.07.97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.05M 90 TPM

07-29-1997 13:46

M

h. Lecture

Spectrophotométrie

GAL 233

Chromomètre GAL AL

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0011_1
290.0	3	0.0018_1
290.0	4	0.0014_1
290.0	5	0.0016_1
290.0	6	0.0020_1
290.0	7	0.0015_1
290.0	8	0.0013_1
290.0	9	1.1639_1
290.0	10	1.1791_1
290.0	11	1.1821_1
290.0	12	1.1714_1
290.0	13	1.1672_1
290.0	14	1.1716_1
290.0	15	1.2742_1
290.0	16	1.2897_1
290.0	17	1.2939_1
290.0	18	1.2867_1
290.0	19	1.2769_1
290.0	20	1.2985_1
290.0	21	1.3134_1
290.0	22	1.3265_1
290.0	23	1.3315_1
290.0	24	1.3268_1
290.0	25	1.3158_1
290.0	26	1.3413_1
290.0	27	1.3312_1
290.0	28	1.3482_1
290.0	29	1.3580_1
290.0	30	1.3499_1
290.0	31	1.3318_1
290.0	32	1.3586_1
290.0	33	1.3337_1
290.0	34	1.3506_1
290.0	35	1.3523_1
290.0	36	1.3558_1
290.0	37	1.3375_1
290.0	38	1.3632_1
290.0	39	1.3353_1
290.0	40	1.3545_1
290.0	41	1.3539_1
290.0	42	1.3572_1
290.0	43	1.3382_1
290.0	44	1.3598_1
290.0	45	1.3320_1
290.0	46	1.3509_1
290.0	47	1.3533_1
290.0	48	1.3593_1
290.0	49	1.3387_1
290.0	50	1.3668_1

FOURNIER 1001869

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Subject to  
Protective Order

&amp; 08/08/97

## DISSOLUTION

m:\commun\qlng\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT

DATE : 29/07/97

APPAREIL : GAL 233 GAL 091

LONGUEUR D'ONDE : 290 nm

CUVE en mm: 2

TITRE : LF 178 TER lot RG 2395/01 14 kg LSNa 0,05M 90 tpm

N° CAHIER : LF 178 TER n°2 p 163

FICHER : M:\commun\ing\donnbase\L178ter\dissolution\Not RG 2395-01 14 kg 0,05M 90tpm  
ELUANT : LSNa 0.05M

ELUANT: LSNa 0,05 M

AGITATION : 90 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	160
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

**FOURNIER 1001870**

**Highly Confidential  
Subject to  
Protective Order**

Témoin 100mg/l	0,900
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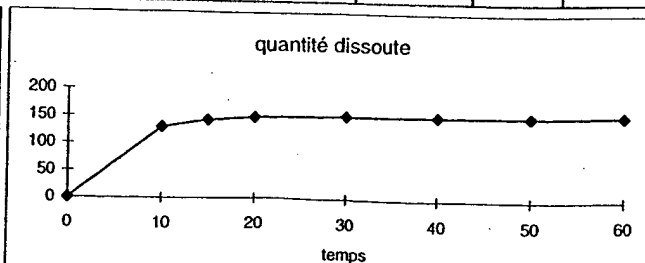
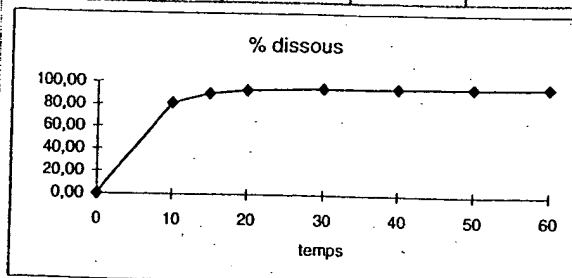
### SUIVI DE LA DISSOLUTION

volumé prélevé en ml		5					
TEMPS	volumé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	1,164	1,179	1,182	1,171	1,167	1,172
15	1000	1,274	1,29	1,294	1,287	1,277	1,299
20	1000	1,313	1,327	1,332	1,327	1,316	1,341
30	1000	1,331	1,348	1,358	1,35	1,332	1,359
40	1000	1,334	1,351	1,352	1,356	1,338	1,363
50	1000	1,335	1,355	1,354	1,357	1,338	1,36
60	1000	1,332	1,351	1,353	1,359	1,339	1,367

### RESULTATS EN % DISSOUS

[illegible]

### RESULTATS EN QUANTITE DISSOLTE

[illegible]



29.07.97

Dissolution. LF 126 ter

166

Lot TG 132 - 18 kg.

L3Na 0,025N. 75TPN

## 1. Préparation du milieu de dissolution Bal. GALCOS

Pesée de l'eau

EAU

29.07.97 14:51:16

0.00 g

448.21 g Tax

0.00 g

3462.4 g Net

0.00 g

3910.6 g Brut

0.00 g

448.59 g Tax

0.00 g

3507.2 g Net

0.00 g

3955.8 g Brut

0.00 g

448.45 g Tax

0.00 g

3509.7 g Net

0.00 g

3958.2 g Brut

0.00 g

448.45 g Tax

0.00 g

2482.5 g Net

0.00 g

2938.9 g Brut

o quantité d'eau pesée

$$3462,4 + 3507,2 + 3509,7 + 2482,5 = 12961,8 \text{ g.}$$

o Pour un milieu à 0,025N, quantité de L3Na ci peser

$$\frac{12961,8}{1000} \times 288,4 \times 0,025 = 93,5 \text{ g.}$$

L3Na

29.07.97 15:14:23  
Code ARR 1547

0.00 g

143.22 g Tax

0.00 g

93.50 g Net

~~93.50 g~~ M

0.00 g

236.72 g Brut

M

FOURNIER 1001871

Highly Confidential  
Subject to  
Protective Order

22.08/08/97

167

2. Remplissage des bols de dissolution. Bal GAL 065

1 litre de LNa 0.025N pese 1001.0 g.

29.07.97

15:48:04

1 0.00 g

761.1 g Tare

0.00 g

1001.0 g Net

0.00 g

1762.1 g Brut

3 0.00 g

769.0 g Tare

0.00 g

1001.0 g Net

0.00 g

1770.0 g Brut

5 0.00 g

767.1 g Tare

0.00 g

1001.0 g Net

0.00 g

1768.1 g Brut

2 0.00 g

760.6 g Tare

0.00 g

1001.0 g Net

0.00 g

1761.6 g Brut M

4 0.00 g

699.0 g Tare

0.00 g

1001.0 g Net

0.00 g

1700.0 g Brut M

6 0.00 g

753.2 g Tare

0.00 g

1001.0 g Net

0.00 g

1754.2 g Brut M

3. Pesée des comprimés Bal GAL 805

LF 77812

29.07.97

15:31:03

Code Lot: TG 192-18 kg.

1

0.0 mg

697.9 mg

2

0.0 mg

689.4 mg

3

0.0 mg

690.5 mg

4

0.0 mg

705.8 mg

5

0.0 mg

702.9 mg

6

0.0 mg

691.9 mg

h. Conditions

Dissolution GAL 091

FOURNIER 1001872

Highly Confidential  
Subject to  
Protective Order

T° 37°C ± 0.5 à 30/07/97 M 30.07.97

Q 75 TPA à 30/07/97 M 30.07.97

S. Cecture

Spectrophotomètre GAZ 222

Chromomètre GAZ 122

168

LF 178 TER LOT TG 192 18 KG LSNA 0.025M 75 TPM

07-30-1997 10:15

M

Lambda	No.	Valeur_E
290.0	1	-0.0000_1
290.0	2	-0.0004_1
290.0	3	0.0019_1
290.0 <sup>0</sup>	4	0.0014_1
290.0	5	0.0003_1
290.0	6	0.0003_1
290.0	7	0.0007_1
290.0	8	0.0007_1
290.0	9	0.7182_1
290.0 <sup>10'</sup>	10	0.8995_1
290.0	11	0.9057_1
290.0	12	0.7102_1
290.0	13	0.7481_1
290.0	14	0.9140_1
290.0	15	1.0507_1
290.0 <sup>15'</sup>	16	1.1066_1
290.0	17	1.0954_1
290.0	18	1.0424_1
290.0	19	1.0472_1
290.0	20	1.0857_1
290.0	21	1.1993_1
290.0 <sup>20'</sup>	22	1.2054_1
290.0	23	1.1904_1
290.0	24	1.2016_1
290.0	25	1.1876_1
290.0	26	1.1783_1
290.0	27	1.3212_1
290.0 <sup>30'</sup>	28	1.2848_1
290.0	29	1.2707_1
290.0	30	1.3301_1
290.0	31	1.3082_1
290.0	32	1.2581_1
290.0	33	1.3948_1
290.0 <sup>40'</sup>	34	1.3213_1
290.0	35	1.3111_1
290.0	36	1.3851_1
290.0	37	1.3637_1
290.0	38	1.2969_1
290.0	39	1.3996_1
290.0 <sup>50'</sup>	40	1.3385_1
290.0	41	1.3243_1
290.0	42	1.4046_1
290.0	43	1.3894_1
290.0	44	1.3128_1
290.0	45	1.4190_1
290.0 <sup>60'</sup>	46	1.3533_1
290.0	47	1.3400_1
290.0	48	1.4224_1
290.0	49	1.4080_1
290.0	50	1.3250_1
290.0	51	1.4633_1
290.0 <sup>120'</sup>	52	1.3897_1
290.0	53	1.3839_1
290.0	54	1.4677_1
290.0	55	1.4606_1
290.0	56	1.3728_1

FOURNIER 1001873

Highly Confidential  
Subject to  
Protective Order

2 08/08/92

## DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

## CONDITIONS ET MODÈS OPERATOIRES

OPERATEUR : D.LECRIT  
 DATE : 30/07/97  
 APPAREIL : GAL 233 GAL 091  
 LONGUEUR D'ONDE : 290 nm  
 CUVE en mm: 2

TITRE : LF 178 TER lot TG 192 18 kg LSNa 0,025M 75 tpm  
 N° CAHIER : LF 178 TER n°2 p 166  
 FICHER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot TG 192 18 kg 0,025M 75tpm  
 ELUANT : LSNa 0,025 M  
 AGITATION : 75 TPM

## PREPARATION DES ECHANTILLONS

masse théorique	694,4
dosage théorique	160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	697,90	689,40	690,50	705,80	702,90	691,90
quantité de principe actif	160,81	158,85	159,10	162,63	161,96	159,42

FOURNIER 1001874

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Témoin 100mg/l 0,900

## SUIVI DE LA DISSOLUTION

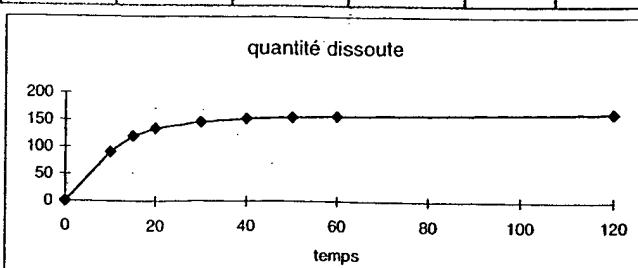
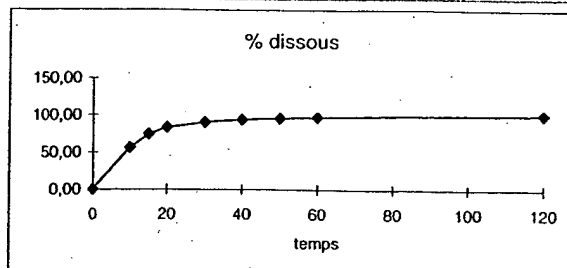
TEMPS	volume en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,718	0,9	0,906	0,71	0,748	0,914
15	1000	1,051	1,107	1,095	1,042	1,047	1,086
20	1000	1,199	1,205	1,19	1,202	1,188	1,178
30	1000	1,321	1,285	1,271	1,33	1,308	1,258
40	1000	1,395	1,321	1,311	1,385	1,364	1,297
50	1000	1,4	1,339	1,324	1,405	1,389	1,313
60	1000	1,419	1,353	1,34	1,422	1,408	1,325
120	1000	1,463	1,39	1,384	1,468	1,461	1,373

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	56,56	49,61	62,95	63,27	48,51	51,32	63,70	7,45	13,17
15,0	74,49	72,87	77,75	76,79	71,43	72,09	76,01	2,68	3,60
20,0	83,31	83,46	84,99	83,80	82,72	82,12	82,80	1,01	1,21
30,0	90,77	92,30	91,01	89,88	91,88	90,76	88,78	1,29	1,43
40,0	94,67	97,87	93,97	93,11	96,09	95,05	91,94	2,13	2,25
50,0	96,26	98,70	95,70	94,48	97,93	97,23	93,51	2,04	2,12
60,0	97,85	100,49	97,14	96,06	99,57	99,01	94,80	2,20	2,25
120,0	101,46	104,03	100,20	99,60	103,20	103,13	98,61	2,26	2,23
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	90,67	79,78	100,00	100,67	78,89	83,11	101,56	11,14	12,28
15,0	119,49	117,18	123,50	122,17	116,17	116,75	121,17	3,16	2,65
20,0	133,68	134,21	135,00	133,33	134,53	133,00	132,00	1,11	0,83
30,0	145,66	148,43	144,56	143,00	149,42	146,99	141,54	3,12	2,15
40,0	151,93	157,38	149,28	148,15	156,27	153,94	146,58	4,53	2,98
50,0	154,48	158,71	152,01	150,32	159,26	157,48	149,07	4,53	2,93
60,0	157,03	161,60	154,31	152,83	161,93	160,36	151,14	4,81	3,06
120,0	162,83	167,28	159,17	158,47	167,83	167,03	157,21	5,03	3,09
0,0									
0,0									
0,0									
0,0									



30.07.97

Dissolution LF 178ter

170

Lot RG 2395/01. 14 kg.

L3Nor 0,025N - 120 TPN

1. Préparation des milieux de dissolution.

Voir page 166

2. Remplissage des bts de dissolution - Bal. GAL 065

1 litre de L3Nor 0,025N pèse 1001,0 g.

30.07.97 10:47:20

1 0.00 g  
753.3 g Tare  
0.00 g  
1001.0 g Net -  
0.00 g  
1754.3 g Brut

2 0.00 g  
699.2 g Tare  
0.00 g  
1001.0 g Net -  
0.00 g  
1700.2 g Brut M

3 0.00 g  
760.8 g Tare  
0.00 g  
1001.0 g Net -  
0.00 g  
1761.8 g Brut

4 0.00 g  
761.1 g Tare  
~~-0.01 g~~  
0.00 g  
1001.0 g Net -  
0.00 g  
1762.1 g Brut M

5 0.00 g  
767.3 g Tare  
0.00 g  
1001.0 g Net -  
0.00 g  
1768.3 g Brut

6 0.00 g  
769.2 g Tare  
0.00 g  
1001.0 g Net -  
0.00 g  
1770.2 g Brut M

3. Pesée des comprimés Bal. GAL 205

FOURNIER 1001875

Highly Confidential  
Subject to  
Protective Order

LF 178ter  
30.07.97 10:08:22  
Code RG 2395/01-14 kg  
ID 1 0.0 mg  
ID 2 702.0 mg  
ID 3 0.0 mg  
ID 4 697.0 mg  
ID 5 0.0 mg  
ID 6 704.5 mg  
ID 7 0.0 mg  
ID 8 700.0 mg  
ID 9 0.0 mg  
ID 10 701.0 mg  
ID 11 0.0 mg  
ID 12 703.5 mg

30 08/08/97

171

h. Conditions

Dissoltest GAL 081

T = 37°C ± 0.5 &amp; 30/07/92 M 30.07.97

C = 120 TPM &amp; 30/07/92 M 30.07.97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.025M 120 TPM

M 07-30-1997 14:36

Lecture

Spectrophotométrie GAL 233

Chronométré GAL 122

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0004_1
290.0	3	0.0003_1
290.0	4	0.0009_1
290.0	5	0.0005_1
290.0	6	0.0004_1
290.0	7	0.0003_1
290.0	8	0.0003_1
290.0	9	0.9735_1
290.0	10	0.9939_1
290.0	11	0.9749_1
290.0	12	0.9761_1
290.0	13	0.9937_1
290.0	14	0.9847_1
290.0	15	1.1292_1
290.0	16	1.1361_1
290.0	17	1.1359_1
290.0	18	1.1315_1
290.0	19	1.1397_1
290.0	20	1.1487_1
290.0	21	1.2099_1

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30 08/08/97

T = 37°C ± 0,5    & 30/07/92 M 30.07.9  
 O = 120 TPM    & 30/07/92 M 30.07.9

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.025M 120 TPM

M 07-30-1997 14:36

Lecture

Spectrophotométre GAL 233  
 Chronométre GAL 122

Lambda	No.	Valeur_E
290.0	1	0.0000_1
290.0	2	0.0004_1
290.0	3	0.0003_1
290.0	4	0.0009_1
290.0	5	0.0005_1
290.0	6	0.0004_1
290.0	7	0.0003_1
290.0	8	0.0003_1
290.0	9	0.9735_1
290.0	10	0.9939_1
290.0	11	0.9749_1
290.0	12	0.9761_1
290.0	13	0.9937_1
290.0	14	0.9847_1
290.0	15	1.1292_1
290.0	16	1.1361_1
290.0	17	1.1359_1
290.0	18	1.1315_1
290.0	19	1.1397_1
290.0	20	1.1487_1
290.0	21	1.2099_1
290.0	22	1.2086_1
290.0	23	1.2162_1
290.0	24	1.2140_1
290.0	25	1.2149_1
290.0	26	1.2283_1
290.0	27	1.2698_1
290.0	28	1.2698_1
290.0	29	1.2789_1
290.0	30	1.2774_1
290.0	31	1.2741_1
290.0	32	1.2878_1
290.0	33	1.2966_1
290.0	34	1.2938_1
290.0	35	1.3072_1
290.0	36	1.3000_1
290.0	37	1.2969_1
290.0	38	1.3143_1
290.0	39	1.3064_1
290.0	40	1.3017_1
290.0	41	1.3121_1
290.0	42	1.3121_1
290.0	43	1.3077_1
290.0	44	1.3280_1
290.0	45	1.3123_1
290.0	46	1.3072_1
290.0	47	1.3166_1
290.0	48	1.3149_1
290.0	49	1.3099_1
290.0	50	1.3307_1
290.0	51	1.3276_1
290.0	52	1.3257_1
290.0	53	1.3352_1
290.0	54	1.3301_1
290.0	55	1.3280_1
290.0	56	1.3505_1

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# DISSOLUTION

m:\commun\glnq\traitdon\distem5

date édition: le 06/02/97

172

## CONDITIONS ET MODES OPERATOIRES

OPERATEUR : D.LECRIT  
DATE : 30/07/97  
APPAREIL : GAL 233 GAL 091  
LONGUEUR D'ONDE : 290 nm  
CUVE en mm : 2

TITRE : LF 178 TER lot RG 2395/01 14 kg LNa 0,025M 120 tpm  
N° CAHIER : LF 178 TER n°2 p 170  
FICHIER : M:\commun\glnq\donnbase\LF178ter\dissolution\lot RG 2395-01 14 kg 0,025M 120tpm  
ELUANT : LNa 0,025 M  
AGITATION : 120 TPM

## PREPARATION DES ECHANTILLONS

masse théorique 160  
dosage théorique 160 en mg

	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
masse de la prise d'essai	160,00	160,00	160,00	160,00	160,00	160,00
quantité de principe actif	160,00	160,00	160,00	160,00	160,00	160,00

Témoin 100mg/l 0,900

Fournier 1001878

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## SUIVI DE LA DISSOLUTION

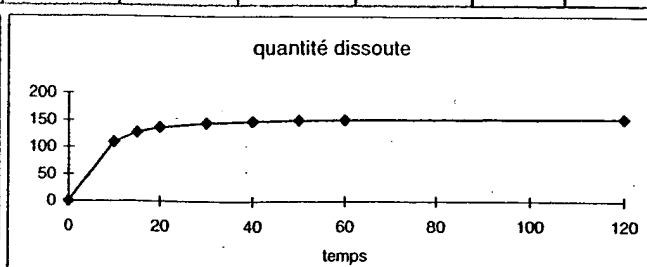
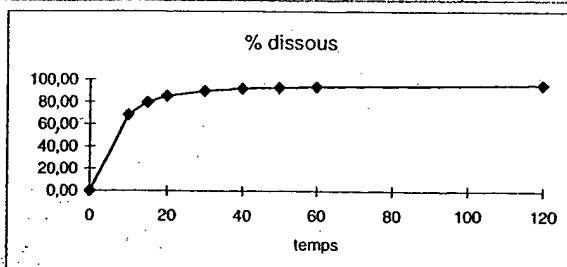
TEMPS	volume prélevé en ml	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6
0	0	0	0	0	0	0	0
10	1000	0,974	0,994	0,975	0,976	0,994	0,985
15	1000	1,129	1,136	1,136	1,132	1,14	1,149
20	1000	1,21	1,209	1,216	1,214	1,215	1,228
30	1000	1,27	1,27	1,279	1,277	1,274	1,288
40	1000	1,297	1,294	1,307	1,3	1,297	1,314
50	1000	1,306	1,302	1,312	1,312	1,308	1,328
60	1000	1,312	1,307	1,317	1,315	1,31	1,331
120	1000	1,328	1,326	1,335	1,33	1,328	1,351

## RESULTATS EN % DISSOUS

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0,00	0	0	0	0	0	0		
10,0	68,26	67,64	69,03	67,71	67,78	69,03	68,40	0,65	0,95
15,0	79,30	78,74	79,23	79,23	78,95	79,51	80,13	0,49	0,61
20,0	85,13	84,76	84,70	85,18	85,04	85,12	86,02	0,47	0,56
30,0	89,79	89,34	89,35	89,97	89,83	89,64	90,61	0,47	0,53
40,0	91,98	91,66	91,46	92,36	91,87	91,67	92,86	0,53	0,58
50,0	93,12	92,74	92,47	93,16	93,16	92,89	94,29	0,63	0,68
60,0	93,85	93,61	93,27	93,97	93,82	93,48	94,96	0,60	0,64
120,0	95,53	95,17	95,04	95,67	95,32	95,19	96,81	0,66	0,69
0,0									
0,0									
0,0									
0,0									

## RESULTATS EN QUANTITE DISSOUE

TEMPS	MOYENNE	CELLULE 1	CELLULE 2	CELLULE 3	CELLULE 4	CELLULE 5	CELLULE 6	écart type	CV
0,0	0	0	0	0	0	0	0		
10,0	109,22	108,22	110,44	108,33	108,44	110,44	109,44	1,04	0,95
15,0	126,88	125,99	126,77	126,76	126,32	127,22	128,21	0,78	0,61
20,0	136,21	135,61	135,52	136,28	136,06	136,19	137,63	0,76	0,56
30,0	143,67	142,95	142,97	143,96	143,73	143,42	144,98	0,76	0,53
40,0	147,17	146,66	146,34	147,78	147,00	146,68	148,58	0,85	0,58
50,0	148,99	148,38	147,95	149,06	149,06	148,62	150,87	1,01	0,68
60,0	150,16	149,77	149,23	150,35	150,12	149,57	151,94	0,96	0,64
120,0	152,86	152,28	152,06	153,08	152,51	152,30	154,90	1,06	0,69
0,0									
0,0									
0,0									
0,0									



52 08/08/98



Mr. 30.07.017

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M 30.07.97

FOURNIER 1001880

Highly Confidential  
Subject to  
Protective Order

M 30 07 97

FOURNIER 1001881

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Subject to  
Protective Order



STATE OF NEW YORK           )  
  )  
  )  
COUNTY OF NEW YORK       )

SS

**CERTIFICATION**

This is to certify that the attached translation is, to the best of my knowledge and belief, a true and accurate translation from French into English of "LF 178TER Lab Notebook, Dissolution No. 1."

Randon Burns, Vice President  
Geotext Translations, Inc.

Sworn to and subscribed before me

this 7<sup>th</sup> day of October, 20 04.

**EZECHIEL A. COPIC**  
Notary Public, State of New York  
No. 01C06104132  
Qualified in Queens County  
Commission Expires January 12, 2008

LF 178TER LAB NOTEBOOK  
DISSOLUTION NO. 1

Drug Formulations Department

Dissolution Lab Notebook No. 1

LF 178 TER

Notebook approved on 04/26/00 by OTT  
[initials] 04/26/00

Notebook begun 02/18/97  
Completed May 15, 1997

02/18/97

Dissolution Tests  
2% Tween 80

I Spectra - Set of standards

A - Preparation of 2% Tween 80 solution

Using a graduated pipette, introduce 40 ml of Tween 80 Prolabo code 28 830 291 lot 045FC into a 200 ml volumetric flask, and qs with the day's purified water.

B - Preparation of the set of standards

With Fenofibrate/sodium lauryl sulfate co-micronizate - ARR 1709

Weigh the co-micronizate in a 200-ml volumetric flask and qs with the 2% Tween 80 solution.

Do the set of standards twice with fenofibrate concentrations of 200 - 150 - 100 - 50 mg/l.

Weighed with balance	GAL205 - AG204
Ultrasound	GAL212
Magnetic stirrer	GAL170

After creating the solution - 15 min ultrasound  
stir overnight in magnetic stirrer.

Fenofibrate concentration (mg/L)	200 mg/l	150 mg/l	100 mg/l	50 mg/l
Weight of co-micronizate	207 mg	155.25 mg	103.5 mg	51.75 mg/l

[initials] 3/6/97

C. Spectral scans

Uvikon 930 - GAL 108      Procedure: GAL051GAL002UFR03

Baseline saved = diskette Product Lip200gr No. 2

water/water: LBEAU [=water baseline]

2% Tween 80/2% Tween 80: LBTWEEN [=Tween baseline]

Spectrum graph

ref: water

standard: 2% Tween 80

SPECTRAL SCAN WATER/TWEEN 80
------------------------------

02/18/1996 4:13 PM

[See original for graph and data.]

$\lambda$  in nm

OD

[initials]

Corresponding  
data:

[See original for data.]

[initials]



01/19/97

The solutions prepared on 02/18/97 for the set of standards are unusable for this purpose, as the quantities weighed were for 1000 ml and not 200 ml, an error of a factor of 5. However, these solutions can be used to determine the saturation concentration for the fenofibrate in the 20% Tween 80 solution.

02/18/97

2:07:21 PM

Fenofibrate concentration in solution A

[See original for data.]

Fenofibrate concentration in solution B

[See original for data.]

Obtaining spectra for 2 filtered solutions compared to water and 2% Tween 80.

Filters used: Millex HA filters, reference SL HA 025 NB. The [illegible] will be calculated after the set of standards is obtained.

[initials] 3/6/9[text cut off]

SOLUTION A COMPARED TO WATER

02/19/1996 2:26 PM

[See original for graph and data.]

A/water

SOLUTION A COMPARED TO TWEEN 80

[date and time obscured]

[See original for graph and data.]

A/Tween 80

[initials] [date obscured]

SOLUTION A COMPARED TO WATER

02/19/1996 2:26 PM

[See original for graph.]

SOLUTION B COMPARED TO TWEEN 80

02/19/1996 2:38 PM

[See original for graph.]

SOLUTION B COMPARED TO WATER

02/19/1996 2:5 [text cut off] PM

[See original for graph and data.]

B/water

[text obscured]

02/19/1996 2:4 [text cut off] PM

[See original for graph and data.]

B/Tween 80

SOLUTION B COMPARED TO WATER

02/19/1996 2: [text cut off] PM

[See original for graph.]

[text obscured]

02/19/1996 2: [text cut off] PM

[See original for graph and data.]

SOLUTION B COMPARED TO WATER

02/19/1996 2: [text cut off] PM

[See original for graph and data.]

SOLUTION B COMPARED TO TWEEN 80

02/19/1996 2: [text cut off] PM

[See original for graph.]

02/19/97

D. Preparation of new solutions for the set of standards

raw materials:

- co-micronizate of fenofibrate/Na lauryl sulfate: ARR 1709,

medium used:

- 2% Tween 80 Prolabo code 28 830 291 lot 045 FC

The co-micronizate are weighed directly in the 200 ml flasks. Then qs to 200 ml with the 2% Tween 80. The standards are prepared twice for feno concentrations: 50 mg/l - 100 mg/l - 150 mg/l - 200 mg/l.

Utilized balance AG204 GAL205.

Concentration - fenofibrate	Concentration - co-micronizate	Weight of co-micronizate needed for a volume of 200 ml
[See original for data.]		

Use 2% Tween 80 prepared on 02/18/97, and prepare a new liter of solution.

Use a graduated pipette to introduce 20 ml of Tween 80 Prolabo code 28 830 291 into a 1000 ml volumetric flask. Qs with the day's purified water. Stir for 1 hour.

The solutions are left overnight in the stirrer.

Calculating concentrations  
for solutions A and B

[See original for data.]

[initials] 3/6/97



02/20/97

D. Obtaining spectra for each solution compared to water and to 2% Tween 80.

Note: will use the 50, 100, and 150 mg/l solutions to determine the set of standards; the 200 mg/l solution will be used to confirm the saturation concentration because it is still not clear after 1 night of stirring and 15 minutes of ultrasound.

50 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 9:5 [text cut off] AM

50 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 9:5 [text cut off] AM

[See original for graph and data.]

[illegible]

[initials] 3/6/97

02/20/97

D. Obtaining spectra for each solution compared to water and to 2% Tween 80.

Note: will use the 50, 100, and 150 mg/l solutions to determine the set of standards; the 200 mg/l solution will be used to confirm the saturation concentration because it is still not clear after 1 night of stirring and 15 minutes of ultrasound.

50 MG/L SOLUTION A COMPARED TO WATER
--------------------------------------

02/20/1996 9: [text cut off] AM

[See original for graph.]

[text cut off]

2/20/97

Obtaining spectra for each solution compared to water and to 2% Tween 80.

Note: will use the 50, 100, and 150 mg/l solutions to determine the set of standards; the 200 mg/l solution will be used to confirm the saturation concentration because it is still not clear after 1 night of stirring and 15 minutes of ultrasound.

50 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 9:5 [text cut off] AM

[See original for graph and data.]

50 mg/A/water

[text cut off]

02/20/97

Obtaining spectra for each solution compared to water and to 2% Tween 80.

Note: will use the 50, 100, and 150 mg/l solutions to determine the set of standards; the 200 mg/l solution will be used to confirm the saturation concentration because it is still not clear after 1 night of stirring and 15 minutes of ultrasound.

50 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 9:5 [text cut off] AM

50 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 9:5 [text cut off] AM

[See original for graph.]

[initials] 3/6/97

50 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:13 AM

50 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:07 AM

[See original for graph and data.]

[illegible] B/Tween

100 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 10:21 AM

100 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 10:25 AM

[See original for graph and data.]

100 mg A/Tween

50 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:1 [text cut off] AM

[See original for graph and data.]

50 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:1 [text cut off] AM

[See original for graphs and data.]

50 mg B/water

[illegible]

50 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:1 [text cut off] AM

50 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:0 [text cut off] AM

[See original for graph.]

100 MG/L SOLUTION A COMPARED TO WATER

100 MG/L SOLUTION A COMPARED TO TWEEN 80

[date and time obscured]

[See original for graph and data.]

100 mg A/water



50 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:1 [text cut off] AM

50 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:0 [text cut off] AM

[See original for graph and data.]

[illegible] B/Tween

100 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 10:2 [text cut off] AM

[See original for graph.]

50 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:13 AM

50 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:07 AM

[See original for graph and data.]

[illegible] B/Tween

100 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 10:21 AM

[See original for graph and data.]

100 mg A / water

50 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:1 [text cut off] AM

50 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:0 [text cut off] AM

[See original for graph and data.]

[illegible] B/Tween

100 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 10:2 [text cut off] AM

100 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 10:2 [text cut off] AM

[See original for graph.]

50 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:1 [text cut off] AM

50 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:0 [text cut off] AM

[See original for graph and data.]

[illegible] B/Tween

100 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 10:2 [text cut off] AM

100 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 10:2 [text cut off] AM

[See original for graph and data.]

100 mg A/Tween

sticking error  
2/21/97  
[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:59 AM

100 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:45 AM

[See original for graph and data.]

100 B/Tween

150 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:03 AM

150 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 11:10 AM

[See original for graph and data.]

150 mg A/Tween

sticking error  
2/21/97  
[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:59 AM

[See original for graphs and data.]

[illegible]

sticking error

2/21/97

[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:59 AM

[See original for graphs and data.]

100 B/water

[illegible]

sticking error

2/21/97

[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:59 AM

100 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:45 AM

[See original for graph.]

150 MG/L SOLUTION A COMPARED TO WATER

[date and time obscured]

150 MG/L SOLUTION A COMPARED TO TWEEN 80

[date and time obscured]

[See original for graph and data.]

150 mg A/Tween



sticking error  
2/21/97  
[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:5 [text cut off] AM

100 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:4 [text cut off] AM

[See original for graph and data.]

100 B/Tween

150 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:0 [text cut off] AM

150 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 11:1 [text cut off] AM

[See original for graph and data.]

150 mg A/Tween

sticking error  
2/21/97  
[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:5 [text cut off] AM

100 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:4 [text cut off] AM

[See original for graph and data.]

100 B/Tween

150 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:0 [text cut off] AM

[See original for graph.]

sticking error

2/21/97

[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:59 AM

100 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:4 [text cut off] AM

[See original for graph and data.]

100 B/Tween

150 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:03 AM

[See original for graph and data.]

150 A/water

sticking error  
2/21/97  
[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:59 AM

100 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:45 AM

[See original for graph and data.]

100 B/Tween

150 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:03 AM

150 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 11:10 AM

[See original for graph.]

sticking error  
2/21/97  
[initials]

100 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 10:59 AM

100 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 10:45 AM

[See original for graph and data.]

100 B/Tween

150 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:03 AM

150 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 11:10 AM

[See original for graph and data.]

150 mg A/Tween

150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:2 [text cut off] AM

150 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 11:19 AM

[See original for graph and data.]

150 B/Tween

200 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:31 AM

200 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 1:3 [text cut off] PM

[See original for graph and data.]

200 A [illegible]

150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:25 AM

[See original for graphs and data.]

200 A [illegible]

150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:2 [text cut off] AM

[See original for graphs and data.]

150 B/water

200 A [illegible]



150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:2[text cut off] AM

150 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 11:1[text cut off] AM

[See original for graph.]

200 MG/L SOLUTION A COMPARED TO WATER

[date and time obscured]

200 MG/L SOLUTION A COMPARED TO TWEEN 80

[date and time obscured]

[See original for graph and data.]

200 A [illegible]

150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:25 AM

150 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 11:19 AM

[See original for graph and data.]

150 B/Tween

200 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:31 AM

200 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 1:31 PM

[See original for graph and data.]

200 A [illegible]

150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:2 [text cut off] AM

150 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 11:1 [text cut off] AM

[See original for graph and data.]

150 B/Tween

200 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:3[text cut off] AM

[See original for graph.]

150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:2 [text cut off] AM

150 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 11:1 [text cut off] AM

[See original for graph and data.]

150 B/Tween

200 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:3 [text cut off] AM

[See original for graph and data.]

200 A/water

150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:2 [text cut off] AM

150 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 11:1 [text cut off] AM

[See original for graph and data.]

150 B/Tween

200 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:3 [text cut off] AM

200 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 1: [text cut off] PM

[See original for graph.]

150 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 11:2[text cut off] AM

150 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 11:1[text cut off] AM

[See original for graph and data.]

150 B/Tween

200 MG/L SOLUTION A COMPARED TO WATER

02/20/1996 11:3[text cut off] AM

200 MG/L SOLUTION A COMPARED TO TWEEN 80

02/20/1996 1:3[text cut off] PM

[See original for graph and data.]

200 A [illegible]

200 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 1:[text cut off] PM

200 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 1:[text cut off] PM

[See original for graph and data.]

00 [illegible]

[initials] 3/6/97

200 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 1:43 PM

[See original for graph.]

[initials] [date cut off]



200 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 1:4[text cut off] PM

[See original for graph.]

200 B/water

[initials] [date cut off]

200 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 1:4 [text cut off] PM

200 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 1:37 PM

[See original for graph.]

[initials] [date cut off]

200 MG/L SOLUTION B COMPARED TO WATER

02/20/1996 1:43 PM

200 MG/L SOLUTION B COMPARED TO TWEEN 80

02/20/1996 1:37 PM

[See original for graph and data.]

00 /B /Tween

[initials] [date cut off]

Standard Curve  
Co-micronizate ARR 1709 in 2% Tween 80

A solutions

Conc. mg/l	at 290 nm	at 297 nm
	[See original for data.]	

B solutions

Conc. mg/l	at 290 nm	at 297 nm
	[See original for data.]	

Standard curve - solutions A

[See original for graph.]

OD

Series 1  
Series 2  
Linear (Series 1)  
Linear (Series 2)

Concentration in mg/l

Standard curve - solutions B

[See original for graph.]

OD

Series 1  
Series 2  
Linear (Series 1)  
Linear (Series 2)

Concentration in mg/l

m\cogetc\feno standard

[initials] [date cut off]

Validation of OD coefficients:

at 290 nm:  $\frac{8.6473}{8.6916} \times 100 = 99.5\%$

the OD coefficient is:  $(8.6473 + 8.6916)/2 = \boxed{8.67}$

at 297 nm:  $\frac{7.7915}{7.8292} \times 100 = 99.5\%$

the OD coefficient is:  $(7.7915 + 7.8292)/2 = \boxed{7.81}$

Calculating the saturation concentration:

from 1000 mg/l solutions  
(results analyzed in comparison to Tween)

from 200 mg/l solutions  
(results analyzed in comparison to Tween)

[See original for table data.]

sol A		sol B		sol A		sol B	
		OD				OD	

02/24/97

Set of standards cont.

Preparation of 2 additional solutions, 75 mg/l and 125 mg/l, to supplement and confirm the set of standards already performed [text cut off, possibly the page no.].

1 - Preparation of medium

2% Tween 80

Tween Prolabo code 28 830 291 lot 045FC

the day's purified water

preparation for the set of standard solutions and for dissolution

<u>water:</u>	tare = 2951.6 g	[initials] 02/24/97	[initials] 02/24/97
	gross = 11169.8 g	[initials] 02/24/97	[initials] 02/24/97
	net = 8218.2 g		

Tween:

d = 1.08      have to add 2% Tween =>

$$2 \rightarrow 100$$

$$x \rightarrow 8218.2 + x$$

$$2(8218.2 + x) = 100x$$

$$2 \times 8218.2 + 2x = 100x$$

$$16436.4 = 98x$$

$$x = 167.72 \Rightarrow \text{need to add 167.72 ml of Tween 80}$$

which is 181.14 g

tare = 11169.8 g	[initials] 02/24/97	[initials] 02/24/97
<del>gross</del> = reset	[initials] 02/24/97	[initials] 02/24/97
net = 181.1 g	[initials] 02/24/97	[initials] 02/24/97

used balance PC16 GAL 111

2. Weighing the raw material

Co-micronizate of Feno/Na lauryl sulfate

ARR 1709

for a Feno concentration of:

75 mg/l

125 mg/l

weight needed of co-micronizate:

77.63 mg/l which is 15.53 mg/200 ml

129.38 mg/l which is 25.88 mg/200 ml

[See original for data.]

The solutions are left to stir overnight.

3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE

A

02/25/1996 1:[text cut off] PM

75 MG/L SOLUTION A FENO – TWEEN BASELINE

B

02/25/1996 2:[text cut off] PM

[See original for graph and data.]

75 /Tween B

125 MG/L SOLUTION A FENO – TWEEN BASELINE

A

125 MG/L SOLUTION A FENO – TWEEN BASELINE

02/25/1996 2:[text cut off] PM

[See original for graph and data.]

125 /Tween B



3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE

A

02/25/1996 1:5[text cut off] PM

[See original for graphs and data.]

3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE

A

02/25/1996 1:55 PM

[See original for graphs and data.]

125 [text obscured]

3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE	A
--	---

02/25/1996 1:55 PM

75 MG/L SOLUTION A FENO – TWEEN BASELINE	B
--	---

02/25/1996 2:01 PM

[See original for graph and data.]

125 MG/L SOLUTION A FENO – TWEEN BASELINE	A
---	---

125 MG/L SOLUTION A FENO – TWEEN BASELINE
---

[date and time obscured]

[See original for graph and data.]

125 [illegible]

3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE

A

02/25/1996 1:55 PM

75 MG/L SOLUTION A FENO – TWEEN BASELINE

B

02/25/1996 2:01 PM

[See original for graph and data.]

75 /Tween B

125 MG/L SOLUTION A FENO – TWEEN BASELINE

A

125 MG/L SOLUTION A FENO – TWEEN BASELINE

[date and time obscured]

[See original for graph and data.]

125 /Tween [illegible]

3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE A

02/25/1996 1:55 PM

75 MG/L SOLUTION A FENO – TWEEN BASELINE B

02/25/1996 2:01 PM

[See original for graph and data.]

75 /Tween B

125 MG/L SOLUTION A FENO – TWEEN BASELINE A

02/25/1996 2:13 PM

[See original for graph.]

3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE A

02/25/1996 1:5[text cut off] PM

75 MG/L SOLUTION A FENO – TWEEN BASELINE B

02/25/1996 2:0[text cut off] PM

[See original for graph and data.]

75 /Tween B

125 MG/L SOLUTION A FENO – TWEEN BASELINE A

02/25/1996 2:13 PM

[See original for graph and data.]

[illegible] Tween A

3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE

A

02/25/1996 1:5[text cut off] PM

75 MG/L SOLUTION A FENO – TWEEN BASELINE

B

02/25/1996 2:0[text cut off] PM

[See original for graph and data.]

75 /Tween B

125 MG/L SOLUTION A FENO – TWEEN BASELINE

A

125 MG/L SOLUTION A FENO – TWEEN BASELINE

02/25/1996 2:[text cut off] PM

[See original for graph.]

3. Obtaining the spectra

75 MG/L SOLUTION A FENO – TWEEN BASELINE A

02/25/1996 1:55 PM

75 MG/L SOLUTION A FENO – TWEEN BASELINE B

02/25/1996 2:01 PM

[See original for graph and data.]

75 /Tween B

125 MG/L SOLUTION A FENO – TWEEN BASELINE A

125 MG/L SOLUTION A FENO – TWEEN BASELINE

02/25/1996 2:19 PM

[See original for graph and data.]

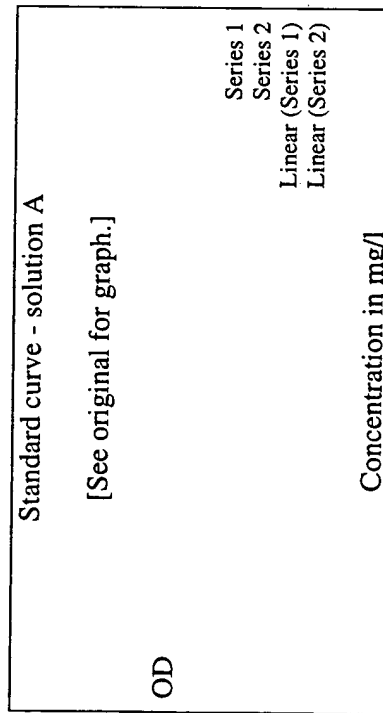
125 /Tween B



Standard Curve  
Co-micronizate ARR 1709 in 2% Tween 80

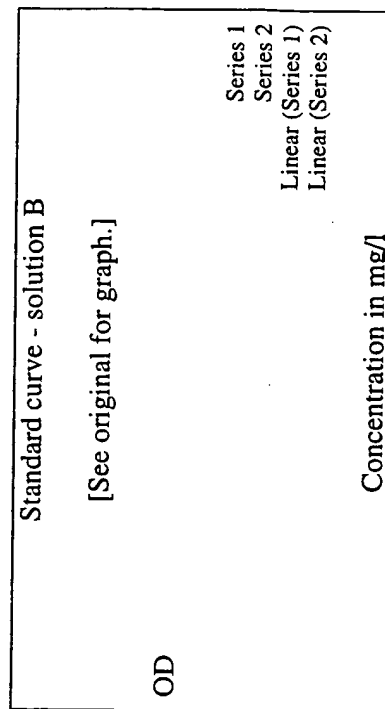
A solutions

Conc. mg/l	at 290 nm	at 297 nm
	[See original for data.]	



B solutions

Conc. mg/l	at 290 nm	at 297 nm
	[See original for data.]	



m\cogetc\feno standard no. 2

Verification of OD coefficients

Calculations at 290 nm:

$$100 \times \frac{8.6125}{8.696} = 99.0\%$$

Use as the OD coefficient:

$$(8.6125 + 8.686)/2 = \boxed{8.65}$$

Calculating the saturation concentration with the OD coefficient of 8.65

1000 mg/l solutions

(using data from page 016)

200 mg/l solutions

at 290 nm	A	B	A	B
	OD = 1530.3	OD = 1530.3	OD = 1504.4	OD = 1490.5
	Cs = 176.9	Cs = 176.9	Cs = 173.9	Cs = 172.[text cut off]

Conclusion:

The saturation concentration [Cs] of fenofibrate in 2% Tween 80 is 176.9 mg/l.

In lab notebook no. 2 for the Lipanthyl 200 tablet, page 98, the saturation concentration of fenofibrate in 0.02M NaLS is 163.6 mg/l.

The 2 Cs values are comparable.

[The entire page is crossed out.]

Dissolution

1) Raw materials

100 mg Fenofibrate tablets  
lot # 340 date 07/02/97 Pharma PASS

2) Preparation of the dissolution medium

see p. 017

3) Weighing the dissolution vessels

1 L of medium

	Reset	Weight	Signature
1			
2			
3			
4			
5			
6			

02/26/97

Preparing a solution  
with ethanol

Preparation identical to the preparation described below.

FEB 24, 1997 10:44 AM PHARMA PASS

+33 3 88 66 35 42 P. 2

Page no: 1/ [illegible]

Analysis no: 1086

[logo:]

PHARMAPASS

Project no: 23 water + 1.25% Tween 80: 1200 ml Lot no: fenofibrate capsules  
23-3 paddle, 50 rpm no. 346  
 $\theta = 0.2$  cm

Control: dissolve approx. 28 mg of fenofibrate (no.313) in  
50 ml of absolute alcohol. Dilute 5/50 ml  
with water + 1.25% Tween 80 [illegible symbol,  
possibly ( $\geq$ )] approx. 56 mg/l

Filters 0.7  $\mu$ m

X: control: 400.0 - 200.0 nm, pts 201; int 1.00; ord -3.982 - 6.0000 A

Inf: control fenofibrate 66.9 mg/l water+1.25%Tween 80, cell:1.00cm

PEAK MAX: THRESH [illegible]

---

Page no: 1/ [illegible] Analysis no: 1086 [logo:]  
PHARMAPASS

---

Project no: 23 water + 1.25% Tween 80: 1200 ml Lot no: fenofibrate capsules  
23-3 paddle, 50 rpm no. 346  
 $\theta = 0.2$  cm

Control: dissolve approx. 28 mg of fenofibrate (no.313) in  
50 ml of absolute alcohol. Dilute 5/50 ml  
with water + 1.25% Tween 80 [illegible symbol,  
possibly ( $\geq$ )] approx. 56 mg/l

Filters 0.7  $\mu$ m

X: control: 400.0 - 200.0 nm, pts 201; int 1.00; ord -3.982 - 6.0000 A  
Inf: control fenofibrate 66.9 mg/l water+1.25%Tween 80, cell:1.00cm  
PEAK MAX: THRESH [illegible]

[See original for table.]

control -> 66.9 mg/l

X: control: 400.0 - 200.0 nm, pts 201; int 1.00; ord -3.982 - 6.0000 A  
Inf: control fenofibrate 66.9 mg/l water+1.25%Tween 80, cell:1.00cm  
PEAK MIN: THRESH 0.0100

[See original for table.]

X: control: 400.0 - 200.0 nm, pts 201; int 1.00; ord -3.982 - 6.0000 A  
Inf: control fenofibrate 66.9 mg/l water+1.25%Tween 80, cell:1.00cm

[See original for graph.]

9/19/95

[signature]

Date: \_\_\_\_\_

x Preparation of 1.25% Tween

Use a graduated pipette to introduce 12.5 ml of Tween 80 reference no. lot 215GC into a 1L flask, qs with purified water.

x Preparation of 1.25% Tween solution with 1/10 ethanol

into a 100 ml flask, use a two-graduation pipette to introduce 10 ml ethanol, qs with the 1.25% Tween solution.

x Preparation of the feno solution

weigh 28.98 mg of co-micronizate ARR 1709 into a 50 ml flask - qs with the ethanol

[See original for data.]

weighed on balance AG204 GAL205

Decant 50 ml of the obtained solution into a 500 ml flask - qs with the 1.25% Tween 80 solution. Homogenize.

02[illegible]  
BASELINE 1.25 PERCENT TWEEN + 1/10 ETHANOL (1CM CUVETTE)

02/26/1996 10:28 AM

[See original for graph.]

BASELINE 56 MG/L FENO IN 1.25 PERCENT TWEEN + 1/10 ETHANOL (1CM CUVETTE)

02/26/1996 10:3[text cut off] AM

[See original for graph.]

Obtained a spectrum which still does not resemble the one obtained by PharmaPass.

02[illegible]

Spectrum for a filtered solution compared to a baseline 2% Tween 80 filtered

75 MG/L SOLUTION A FENO - BASELINE TWEEN FILTERED/TWEEN FILTERED

02/25/1996 10:[text cut off] AM

[See original for graph.]

[text in graph:] still this shoulder



02/26/97

Dissolution in  
2% Tween 80

75 RPM

Tablets lot 340

1. Raw material

100 mg fenofibrate tablets

lot #340. Date 07/02/97 from PharmaPass

2. Preparation of dissolution medium

see page 017

3. Weighing the dissolution vessels

On PC16 GAL111, weighed 1L of 2[text cut off] Tween  
which is 1001.6g (98% at d=1 and 1% at d=1[text cut off])

vessel	reset	weight	signature
1	yes	1001.6 g	[initials] 02/26/97
2	yes	1001.6 g	[initials] 02/26/97
3	yes	1001.6 g	[initials] 02/26/97
4	yes	1001.6 g	[initials] 02/26/97
5	yes	1001.6 g	[initials] 02/26/97
6	yes	1001.6 g	[initials] 02/26/97

4. Weighing the tablets

on balance AG204 GAL205

[See original for data.]

5. Dissolution conditions

dissolutest GAL103

spectro 930 GAL108

{ paddles 75 RPM [initials] 02/26/97  
medium  $37 \pm 0.5^\circ\text{C}$  [initials] 02/26/97  
with 2mm cuvettes  
 $\lambda = 290 \text{ nm}$

Samples collected with 10 ml syringes: 5ml of medium collected and replaced with 5ml of "new" medium.

The syringes are equipped with Prolabo filters ref 178-3985-01.

Readings are taken after filtration with Millex filter SLHA025NB.

Note: the 2% Tween 80 in the reference cuvette is filtered.

6. Measurements

100 MG FENOFIBRATE TABLETS PHARMA PASS LOT 340 2 PERCENT TWEEN 80
---

02/26/1996 3:20 PM

Lambda	No.	Value_E
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[See original for data.]

# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	C. COGET	TITLE	100 mg Fenofibrate tablets Pharma Pass lot 340
DATE	2/26/97	NOTEBOOK NO.	LF 178 TER no. 1
INSTRUMENT	gal 108-gal 103	FILE	m:\commun\ginq\donnbase\LF178TER\Dissolution\340a
WAVELENGTH	290 nm	ELUANT	water + 2% Tween 80
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	100	
theoretical dosage	100	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001510

02/27/97

Dissolution in 2% Tween 80

75 rpm

Lip 200 gelatin capsules ARR1710

1. Raw materials

Lipanthyl 200 gelatin capsules ARR 1710

2. Preparation of dissolution medium

Prolabo Tween reference no. lot 215 GC  
the day's purified water

<u>weighing the</u>	tare = 2953.9 g	[initials] 02/27/97	[initials] 02/27/97
<u>water:</u>	gross = 9754.8 g	[initials] 02/27/97	[initials] 02/27/97
	net = 6800.9 g		

weighing the Tween:

see the calculation on page 17.

weight to be obtained:  $2(6800.9 + x) = 100x$

$2 \times 6800.9 + 2x = 100x$

$x = \frac{13601.8}{98}$

$x = 138.79$

weight to be obtained: 138.79 ml which is 149.9 g ( $138.79 \times 1.08$ )

tare = 9754.8 g	[initials] 02/27/97	[initials] 2/27/97
-----------------	---------------------	--------------------

reset: yes	[initials] 2/27/97
------------	--------------------

net = 149.9 g	[initials] 02/27/97	[initials] 2/27/97
---------------	---------------------	--------------------

Utilization of balance PC16 GAL 111

Stir with overhead stirrer

Highly confidential subject to protective order

FOURNIER 1001511

3. Weighing the gelatin capsules

[See original for weight slip.]

4. Weighing the dissolution vessels

weighed on balance GAL 111.

1L of 2% Tween solution, which is a weight of 1001.6g  
(98% at d=1 and 2% at d=1.08)

vessel	reset	amt weighed	signature
1	yes	1001.6 g	[initials] 2/27/97
2	yes	1001.6 g	[initials] 2/27/97
3	yes	1001.6 g	[initials] 2/27/97
4	yes	1001.6 g	[initials] 2/27/97
5	yes	1001.6 g	[initials] 2/27/97
6	yes	1001.6 g	[initials] 2/27/97

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FOURNIER 1001512

## 5. Dissolution conditions

Prolabo dissolutest GAL 103

paddles 75 rpm 02/27/97 [initials]  
water bath 37°C±0.5 02/27/97 [initials]

UV 930 GAL 108

$\lambda = 290 \text{ nm}$   
cuvettes = 2 mm

Samples collected into the [dissolution] vessels with a 10 m syringe  
5 ml collected and replaced with new medium.

Samples are collected with syringe + tubing [illegible - possibly nozzle]  
+ Prolabo filters ref 178 3985 01.

The OD readings are taken after filtration through Millex filters SLHA025NB.  
The 2% Tween 80 in the reference cuvette is also filtered through a Millex HA filter.

6. Measurements

LIP 200 GELCAPS ARR 1710 IN 2 PERCENT TWEEN 80
--

02/27/1996 11:38 AM

Lambda No. Value\_E

[See original for figures.]

Highly confidential subject to protective order

FOURNIER 1001514



02/26/97

Dissolution of 0.02M NaLS  
1000 ml 75 rpm TABLETS

1. Preparation of dissolution medium

- Purified water weighed on 02/26/97 - Balance GAL014

Tare = 2.725 kg [initials] 02/26/97 [initials] 02/26/97

Gross = 19.550 kg [initials] 02/26/97 [initials] 02/26/97

Gross - Tare = Net = 19.550 - 2.725 = 16,825 kg [initials] 02/26/97  
[initials] 02/26/97

- Weighed NaLS arr1768 - Balance GAL011

[text obscured]

**CLEANING FORM**

**ROOM No:** Balance room

**DATE BEGAN USE OF ROOM:** 02/25/97

**PRODUCT:** Lipanthyl 67 PLACEBO

**LOT No.:** DG 153/00

**OPERATION:** Weighing raw materials

**DATE OPERATION ENDED:** 02/25/97

**ROOM EMPTIED BY:** [initials]

**DATE:** 02/25/97

**EMPTYING OF ROOM VERIFIED BY:** [initials]

**DATE:** 02/26/97

**STATUS:** CONFORMS XX

**DOES NOT CONFORM** \_\_

**CLEANING DONE BY:** [initials]

**DATE:** 02/26/97

**CLEANING VERIFIED BY:** [initials]

**DATE:** 02/26/97

**STATUS:** CONFORMS XX

**DOES NOT CONFORM** \_\_

Highly confidential subject to protective order

FOURNIER 1001515

02/26/97

Dissolution of 0.02M NaLS  
1000 ml - 75 rpm TABLETS

1. Preparation of dissolution medium

- Purified water weighed on 02/26/97 - Balance GAL014

Tare = 2.725 kg	[initials] 02/26/97	[initials] 02/26/97
Gross = 19.550 kg	[initials] 02/26/97	[initials] 02/26/97

Gross - Tare = Net =  $19.550 - 2.725 = 16.825$  kg [initials] 02/26/97  
[initials] 02/26/97

- Weighed NaLS arr1768 - Balance GAL011

$16.825 \times 288.4 \times 0.02M = 97.05g$  of NaLS

SIMPLE WEIGHING

Date weighed: 02/26/1997

04:08:27 PM

\* ARR1768 \*

PRODUCT => NALAUSF

NET WEIGHT => 0.097 KG

TARE => 0.175 KG

GROSS WEIGHT => 0.272 KG

[initials]

Highly confidential subject to protective order

FOURNIER 1001516

2 - Filling the dissolution vessels

Diss [initials]

Balance GAL 111

Prelabo Dissolutest GAL086

1L 0.02M NaLS weighs 1000.0 g

vessel	reset	amt weighed	signature
1	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/[text cut off]
2	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/[text cut off]
3	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/[text cut off]
4	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/[text cut off]
5	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/[text cut off]
6	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/[text cut off]

3 - Weighing the tablets - Balance AG204 - GAL205

[See original for weight slip.]

[initials]

4 - Conditions

Paddles

~~100 rpm~~ [initials]

75 rpm [initials] 02/27/97 [initials] 2/27/97

378±0.5°C [initials] 02/27/97 [initials] 2/27/97

$\lambda$  = 290 nm

UV GAL233

UVIKON 922 33

2mm cuvette

Manual sampling - 5 ml - replaced

Syringe + filter ref 178398501 Filtration through

Millex HA filter ref SLHA025NB prior to reading

Highly confidential subject to protective order

FOURNIER 1001517

FIXED WAVELENGTH 290 NM TABLET 0.02M NALS 75 RPM

[initials]

[See original for figures.]

Lambda No. Value\_E

Air/Air  
NaLS/NaLS

KONTRON INSTRUMENTS UVIKON 922

Operator\_\_\_\_\_

Highly confidential subject to protective order

FOURNIER 1001518

# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	ROSSELIN C	TITLE	Tablets PHARMA PASS LOT 340
DATE	02/27/97	NOTEBOOK NO.	LF 178 TER dissolution no. 1 p. 036
INSTRUMENT	GAL233GAL086	FILE	m:\commun\ginq\donnbase\lf178ter\dissof340b
WAVELENGTH	290 nm	ELUANT	0.02M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	459	
theoretical dosage	100	in mg

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample					
quantity of active substance					

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

[initials]

time

Highly confidential subject to protective order

FOURNIER 1001519

02/26/97

Dissolution of 0.02M NaLS  
1000 ml 75 rpm GELATIN CAPSULES

1. Preparation of dissolution medium

see page 036

2. Filling the dissolution vessels - Balance GAL 111

Balance GAL 111 - Dissolutest AT7 Sotax GAL 091

1L 0.02M NaLS weighs 1000.0g

vessel	reset	amt weighed of 0.02M NaLS	signatures
1	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/97
2	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/97
3	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/97
4	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/97
5	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/97
6	yes	1000.0 g	02/26/97 [initials] [initials] 2/26/97

3 - Weighing the gelatin capsules - Balance AG 204 GAL 205

[See original for weight slip.]

[initials]

4 - Conditions

Paddles 75 rpm [initials] 02/27/97 [initials] 2/27/97  
37°C±0.5°C [initials] 02/27/97 [initials] 2/27/97

Highly confidential subject to protective order

FOURNIER 1001520

[text cut off]

Samples collected manually - 5 ml - replacement - syringe + filter ref. 178398501.

Filtration through Millex filter ref SLHA025NB prior to reading.

FIXED WAVELENGTH 290 NM GELCAP LIP200 ARR1710 0.02M NALS 75 RPM	
---	--

[initials]

[See original for figures.]

[Opérateur = Operator]

Lambda No. Value\_E

Air/Air  
NaLS/NaLS

no. 6=\*error in reading:  
(6 instead of 1)

[initials] 07/[illegible]/97

Highly confidential subject to protective order

FOURNIER 1001521

# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	ROSSELIN C	TITLE	Gelcap LIP 200 plant arr1710
DATE	02/27/97	NOTEBOOK NO.	LF 178TER dissolution no. 1 p. 040
INSTRUMENT	GAL233GAL091	FILE	m:\commun\ginq\donnbase\lf178ter\disso\1710b
WAVELENGTH	290 nm	ELUANT	0.02M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

mass of test sample	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001522



02/28/97

Dissolution of 0.025M NaLS  
Tablets lot 340

75 rpm

1. Raw materials

100 mg fenofibrate tablets

lot #340 date 07/02/97

Pharma Pass

2. Preparation of dissolution medium

purified water of the day:

tare = 3.3922 kg [initials] 02/28/97 [initials] 02/28/97

gross = 11.1991 kg [initials] 02/28/97 [initials] 02/28/97

net = gross - tare = 11.1991 - 3.3922 = 7.8069 kg

NaLS arr1768:

**CLEANING FORM**

**ROOM No:** Balance room

**DATE BEGAN USE OF ROOM:** 02/27/97

**PRODUCT:** LF178TER

**LOT No.:** 2391/00RG

**OPERATION:** Weighing

**DATE OPERATION ENDED:** 02/27/97

**ROOM EMPTIED BY:** [initials]

**DATE:** 02/27/97

**EMPTYING OF ROOM VERIFIED BY:** [initials]

**DATE:** 02/28/97

**STATUS: CONFORMS XX**

**DOES NOT CONFORM**

**CLEANING DONE BY:** [initials]

**DATE:** 02/28/97

**CLEANING VERIFIED BY:** [initials]

**DATE:** 02/28/97

**STATUS: CONFORMS XX**

**DOES NOT CONFORM**

Highly confidential subject to protective order

FOURNIER 1001523

02/28/97

Dissolution of 0.025M NaLS  
Tablets lot 340

75 rpm

1. Raw materials

100 mg fenofibrate tablets  
lot #340 date 07/02/97 Pharm Pass

2. Preparation of dissolution medium

purified water of the day:

tare = 3.3922 g [initials] 02/28/97 [initials] 02/28/97  
gross = 11.1997 g [initials] 02/28/97 [initials] 02/28/97  
net = gross - tare = 11.1991 - 3.3922 = 7.8069 kg

NaLS arr1768:

$7.8069 \times 288.4 \times 0.025 = 56.29$  g to be weighed.

SIMPLE WEIGHING

Date weighed: 02/28/1997

10:26:34 AM

\* ARR1768 \*

PRODUCT =>	NALAUFS
NET WEIGHT =>	0.056 KG
TARE =>	0.170 KG
GROSS WEIGHT =>	0.226 KG

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001524

2. 3. Weighing the dissolution vessels

Weighed on balance GAL 111

1L of 0.025M NaLS which is a weight of 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	[initials] 02/28/97
2	yes	1001.0 g	[initials] 02/28/97
3	yes	1001.0 g	[initials] 02/28/97
4	yes	1001.0 g	[initials] 02/28/97
5	yes	1001.0 g	[initials] 02/28/97
6	yes	1001.0 g	[initials] 02/28/97

4. Weighing the tablets

[See original for weight slip.]

5. Dissolution conditions

Prolabo dissolutest GAL 103

UV 930 GAL 108

[initials] 2/28/97

paddles 75 rpm

$\lambda = 290$  nm

[initials] 02/28/97

water bath  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$

2 mm cuvettes

5 ml sample collected and replaced with new medium

collected with syringe + Prolabo filter ref 17839 8501

assayed after filtration through Millex HA filter  $0.45 \mu\text{m}$

[text cut off]

Highly confidential subject to protective order

FOURNIER 1001525

6. Measurements

TABLETS OF 100 MG FENOFIBRATE PHARMA PASS LOT 340 IN 0.025M NALS

02/28/1996 3:[text cut off] PM

Lambda No. Value\_E

[See original for figures.]

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001526

# DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	C. COGET	TITLE	100 mg Fenofibrate tablets Pharma Pass lot 340
DATE	02/28/97	NOTEBOOK NO.	LF 178 TER no. 1
INSTRUMENT	gal108-gal103	FILE	m:\commun\ging\donbase\LF178TER\Dissolution\340c
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	100	
theoretical dosage	100	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

[initials and date illegible]

time

Highly confidential subject to protective order

FOURNIER 1001527

03/4/97

Dissolution 0.025M NaLS  
Lipanthyl 200M gelcaps Arr1710

1. Preparation of dissolution medium 0.025M NaLS

molecular mass of NaLS = 288.4 g

weight of water:

tare = 2.755 kg [initials] 3/4/97 [initials] 03/4/97

gross = 15.170 kg [initials] 3/4/97 [initials] 03/4/97

net: 15.170 - 2.755 = 12.415 kg

or  $12.415 \times 0.025 \times 288.4 = 89.5$  g of NaLS to be weighed

SIMPLE WEIGHING

Date weighed: 03/04/1997

10:30:29 AM

\* ARR1768 \*

PRODUCT => NALAUSF

NET WEIGHT => 0.090 KG

TARE => 0.264 KG

GROSS WEIGHT => 0.354 KG

2. Weighing the dissolution medium gal 111

1 liter of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signatures
1	yes	1001.0 g	[initials] 03/04/97 [initials]
2	yes	1001.0 g	[initials] 03/04/97 [initials]
3	yes	1001.0 g	[initials] 03/04/97 [initials]
4	yes	1001.0 g	[initials] 03/04/97 [initials]
5	yes	1001.0 g	[initials] 03/04/97 [initials]

[text cut off]

[illegible initials and date]

Highly confidential subject to protective order

FOURNIER 1001528

3. Weighing the gelcaps gal205

[See original for weight slip.]

4. Dissolution conditions

PROLABO dissolutest gal 103

T° = 37°C±0.5°C

[initials] 3/4/97 [initials] 03/4/97

speed = 75 rpm

[initials] 3/4/97 [initials] 03/4/97

5 ml sample collected with replacement of the removed medium by 5 ml of new medium.  
Use of a 10 ml plastic syringe ref Plastipack 302188 equipped with a Prolabo prefilter ref 178398501.

Filtration through Millex HA 0.45 µm ref Millipore SLHA025NB prior to measurement.

5. Measurements

KONTRON 930 spectrophotometer gal 108 with 2mm path length cuvette.

200M LIPANTHYL GELCAPS ARR1710 75 RPM 0.025M NALS

List of parameters

03/04/1996 1:3[text cut off] PM

WAVELENGTH [nm]	290.0
WAIT TIME[s]	0.0
INTEGRATION TIME [s]	5.0
NUMBER OF SAMPLES:	1

CALC MODE	No
-----------	----

CHANGE OF BULBS [nm]	340
DEUTERIUM BULB	Yes
TUNGSTEN BULB	Yes
SLIT(S) [nm]	2.0
AUTO PRINTING	No
AUTO SAVE	No

KONTRON INSTRUMENTS

UVIKON 93

200M LIPANTHYL GELCAPS ARR1710 75 RPM 0.025M NALS

03/04/1996 3:52 PM

Lambda No. Value\_E

AZ [unknown acronym] air/air  
NaLS/NaLS  
AZ [unknown acronym] NaLS/NaLS

[See original for figures.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001530



# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	LIP 200 gelcap plant arr1710
DATE	03/04/97	NOTEBOOK NO.	LF 178TER dissolution no. 1 p 47
INSTRUMENT	gal 103-108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\arr1710c
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

mass of test sample	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials & date illegible]

Highly confidential subject to protective order

FOURNIER 1001531

**COMPARISON OF DISSOLUTIONS FOR PHARMA PASS TABLET  
lot 340 AND THE LIPANTHYL 200M GELCAP ARR 1710**

time	tablets			gelcaps		
	2% Tween 80	0.02M NaLS	0.025M NaLS	2% Tween 80	0.02M NaLS	0.025M NaLS
	[See original for figures.]					

source data from lab notebook LF178ter p27 to p50

tablets

[See original for graph.]

capsules

2% Tween 80  
0.02M NaLS  
0.025M NaLS  
2% Tween 80  
0.02M NaLS  
0.025M NaLS

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001532

3/6/97

Finding the saturation concentration  
of the co-micronizate  
in 0.025M NaLS

Purpose of the test: Find the saturation concentration of the co-micronizate of  
fenofibrate Arr 1709 in 0.025M NaLS.

Protocol: Prepare two solutions of co-micronizate in excess in 0.025M NaLS  
in a 1 liter volumetric flask. Stir for 24 hr gal 119.  
Filter through Millex 0.45  $\mu$ m ref SLHA025NB  
Do a spectral scan using 2mm path length cuvette.  
Reminder: for 100 mg/l of feno absorbance = 0.900

Readings in KONTRON 922 spectrophotometer gal 233  
0.025M NaLS in the reference cuvette  
as per following method.

CHANGE OF BULBS	340
DEUTERIUM LAMP	Yes
TUNGSTEN LAMP	Yes
SLIT(S) [nm]	2.0
AUTOZERO AT STARTUP	No
AUTO PRINTING	No
AUTO SAVE	No
AUTO TRANSFER	No

KONTRON INSTRUMENTS UVIKON 922

Operator\_\_\_\_\_

Highly confidential subject to protective order

FOURNIER 1001533

3/6/97

Finding the saturation concentration  
of the co-micronizate  
in 0.025M NaLS

Purpose of the test: Find the saturation concentration of the co-micronizate of  
fenofibrate Arr 1709 in 0.025M NaLS.

Protocol: Prepare two solutions of co-micronizate in excess in 0.025M NaLS  
in a 1 liter volumetric flask. Stir for 24 hr gal 119.  
Filter through Millex 0.45  $\mu$ m ref SLHA025NB  
Do a spectral scan in 2mm path length cuvette.  
Reminder: for 100 mg/l of feno absorbance = 0.900

[text obscured]

SOLUTION OF CO-MICRONIZATE IN 0.025M NALS SOLUTION B

Parameter list

03/07/1997 09:37 AM

START-END WAVELENGTH [nm]	200-350
SCANNING RATE [nm/min]	200
SAMPLING INTERVAL [nm]	0.5
No. OF CYCLES	1
CYCLE TIME [min]	0.0
NUMBER OF SAMPLES	1
CALC MODE	Peak detection
Peak mode	Peaks
Sensitivity	0.1000 ABS
CHANGE OF BULBS [nm]	340
DEUTERIUM BULB	Yes
TUNGSTEN BULB	Yes
SLIT(S) [nm]	2.0
AUTOZERO AT STARTUP	No
AUTO PRINTING	No
AUTO SAVE	No
AUTO TRANSFER	No

KONTRON INSTRUMENTS UVIKON 922

Operator\_\_\_\_\_

Highly confidential subject to protective order

FOURNIER 1001534

SOLUTION OF CO-MICRONIZATE IN 0.025M NALS SOLUTION A

[See original for graph and figures.]

SOLUTION OF CO-MICRONIZATE IN 0.025M NALS SOLUTION B

[See original for graph and figures.]

Results of peak detection

Sensitivity 0.1000  
Sensitivity mode ABS

Scale 1

Loc  
Value

Peak  
290.50  
1.9681

KONTRON INSTRUMENTS UVIKON 922

Operator \_\_\_\_\_

Solution A: Cs =  $\frac{1.954 \times 100}{0.900} = 217.1$  mg of fenofibrate, or  
 $\frac{217.1 \times 207}{200} = 224.7$  mg of co-micronizate

Solution B => Cs =  $\frac{1.968 \times 100}{0.900} = 218.7$  mg of fenofibrate, or  
 $\frac{218.7 \times 207}{200} = 226.3$  mg of co-micronizate

[illegible date] [initials]

Highly confidential subject to protective order

FOURNIER 1001535

SOLUTION OF CO-MICRONIZATE IN 0.025M NALS SOLUTION A

[See original for graph and figures.]

Results of peak detection

Sensitivity 0.1000  
Sensitivity mode ABS

Scale 1

Loc  
Value

Peak  
290.50  
1.9540

KONTRON INSTRUMENTS UVIKON 922

Operator\_\_\_\_\_

[text obscured]

Highly confidential subject to protective order

FOURNIER 1001536

Result

The saturation concentration for the co-micronizate Arr 1709  
in 0.025M NaLS is  $\frac{(224.7 + 226.3)}{2} = 225.5 \text{ mg}$

225.5 mg/l

that of fenofibrate is  $\frac{217.1 + 218.7}{2} = 217.9 \text{ mg/l}$

07/03/97 [initials]

[date illegible]

Dissolution of tablet  
PharmaPass lot 351

75 Preparation of 0.025M NaLS

molecular mass of NaLS = 288.4 g

weight of water:

tare = 2.785 kg      [initials] 3/10/97      [initials] 03/10/97  
gross = 23.550 kg    [initials] 3/10/97      [initials] 03/10/97  
net: 23.550 - 2.785 = 20.765 kg

which is  $20.765 \times 0.025 \times 288.4 = 149.7$  g of NaLS to be weighed  
see next page for the weight slip

2. Weighing the dissolution medium gal 111  
1L of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	03/10/97 [initials] [initials] 3/10/97
2	yes	1001.0 g	03/10/97 [initials] [initials] 3/10/97
3	yes	1001.0 g	03/10/97 [initials] [initials] 3/10/97
4	yes	1001.0 g	03/10/97 [initials] [initials] 3/10/97
5	yes	1001.0 g	03/10/97 [initials] [initials] 3/10/97
6	yes	1001.0 g	03/10/97 [initials] [initials] 3/10/97

3. Dissolution conditions

dissolutest paddles gal 103

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  \      [initials] 3/10/97      [initials] 03/10/97  
speed = 75 rpm /

5 ml sample collected with replacement of the collected medium with new.  
Utilization of a syringe ref. Plastipak 302188 equipped with a Prolabo prefilter ref.  
178398501.

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001538



SIMPLE WEIGHING

Date weighed: 03/10/1997

09:32:47 AM

75 ARR1768 \*

PRODUCT => NALAUSF

NET WEIGHT => 0.150 KG

TARE => 0.266 KG

GROSS WEIGHT => 0.416 KG

75 Weighing the tablets  
balance AB204 gal 205

1 tablet of 434 mg contains 100 mg of fenofibrate.

[See original for weight slip.]

75 Readings

[initials] 07/03/97

In KONTRON 922 930 spectrophotometer gal 108

filtration of samples through Millex filter ref Millipore SLHA025NB

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001539

# DISSOLUTION

m:\commun\ginq\traidon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	100 mg fenofibrate tablets PHARMA PASS lot 351
DATE	03/10/97	NOTEBOOK NO.	LF 178ter p 55
INSTRUMENT	gal 103 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 351
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	434	
theoretical dosage	100	in mg

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

mass of test sample  
quantity of active substance

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001540

LIP 100 TABLET LOT 351

03/10/1996 3:00 PM

Lambda No. Value\_E

[See original for figures.]

AZ [unknown acronym] air/air

NaLS/NaLS

AZ [unknown acronym] NaLS/NaLS

Highly confidential subject to protective order

FOURNIER 1001541

3/11/97

Dissolution of fenofibrate tablet  
100 mg PHARMA PASS lot 354

These 434 mg tablets contain 100 mg of fenofibrate

1. Preparation of the 0.025M NaLS

see p. 55

2. Weighing the dissolution medium gal 111  
1L of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signature	
1	yes	1001.0 g	[initials] 03/11/97	[initials] 3/11/97
2	yes	1001.0 g	[initials] 03/11/97	[initials] 3/11/97
3	yes	1001.0 g	[initials] 03/11/97	[initials] 3/11/97
4	yes	1001.0 g	[initials] 03/11/97	[initials] 3/11/97
5	yes	1001.0 g	[initials] 03/11/97	[initials] 3/11/97
6	yes	1001.0 g	[initials] 03/11/97	[initials] 3/11/97

3. Weighing the tablets gal 205

[See original for weight slip.]

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001542

4. Dissolution conditions

Prolabo dissolutest with paddles gal 103

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  \\  
speed = 75 rpm / [initials] 3/11/97 [initials] 03/11/97

Collected 5 ml sample of the medium, replaced with new medium.  
Used a 10 ml plastic syringe ref. Plastipack 302188 equipped with a Prolabo prefilter ref. 178398501.

5. Readings [initials] 07/03/97  
in KONTRON 922 930 spectrophotometer gal 108

2mm path length cuvette

TABLET PHARMA PASS 100MG FENO LOT 354
---------------------------------------

03/11/1996 12:41 PM

Lambda	No.	Value_E
--------	-----	---------

		AZ [unknown acronym]
		air/air
		NaLS/NaLS
		AZ [unknown acronym]
		NaLS/NaLS

[See original for figures.]

[initials] 03/07/97

Highly confidential subject to protective order

FOURNIER 1001543

# DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	100 mg fenofibrate tablets PHARM PASS lot 354
DATE	03/11/97	NOTEBOOK NO.	LF 178ter p 58
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 354
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	434	
theoretical dosage	100	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001544

Dissolution of Pharma Pass tablet  
100 mg fenofibrate lot 358

1. Preparation of the dissolution medium NaLS 0.025M

see p. 55

2. Weighing the dissolution medium gal 111

1L of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	[initials] 03/11/97 [initials] 3/11/97
2	yes	1001.0 g	[initials] 03/11/97 [initials] 3/11/97
3	yes	1001.0 g	[initials] 03/11/97 [initials] 3/11/97
4	yes	1001.0 g	[initials] 03/11/97 [initials] 3/11/97
5	yes	1001.0 g	[initials] 03/11/97 [initials] 3/11/97
6	yes	1001.0 g	[initials] 03/11/97 [initials] 3/11/97

3. Weighing the tablets gal 205

1 tablet weighs an average of 434 mg and contains 100 mg of feno

[See original for weight slip.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001545

4. Dissolution conditions

Prolabo dissolutest with paddles gal 103

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$

speed = 75 rpm

\

/ [initials] 3/11/97 [initials] 03/11/97

5 ml sample collected of the medium for measurement, with replacement of the collected medium with 5 ml of new medium.

Utilization of a 10 ml plastic syringe ref. Plastipack 302188 equipped with a Prolabo prefilter ref. 178398501.

5. Readings [initials] 07/03/97

using KONTRON 922 930 spectrophotometer gal 108 in 2mm path length cuvettes

filtration of samples through Millex HA 0.45 $\mu\text{m}$  ref Millex SLHA025NB

TABLET PHARMA PASS 100MG FENO LOT 358
---------------------------------------

03/11/1996 4:07 PM

Lambda	No.	Value_E
--------	-----	---------

		AZ [unknown acronym]
--	--	----------------------

		air/air
--	--	---------

		NaLS/NaLS
--	--	-----------

		AZ [unknown acronym]
--	--	----------------------

		NaLS/NaLS
--	--	-----------

[See original for figures.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001546



[numbers cut off]

290.0

23

0.7620\_1

**DISSOLUTION**

m:\commun\ginq\traidon\distern5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	100 mg fenofibrate tablet PHARMA PASS lot 358
DATE	03/11/97	NOTEBOOK NO.	LF 178ter p 61
INSTRUMENT	gal 103 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 358
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

SAMPLE PREPARATION

theoretical mass	434	
theoretical dosage	100	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001547

3/13/97

Dissolution of Pharma Pass tablet  
100 mg fenofibrate lot 361

1. Preparation of the dissolution medium 0.025M NaLS  
molecular mass of NaLS = 288.4 g

weighing the water:

tare = 2.775 kg	[initials] 3/13/97	[initials] 03/13/97
gross = 20.180 kg	[initials] 3/13/97	[initials] 03/13/97
net 20.180 - 2.775 = 17.405 kg		

which is  $17.405 \times 0.025 \times 288.4 = 125.5\text{g}$  of NaLS to be weighed

SIMPLE WEIGHING

Date weighed: 03/13/1997      11:20:29 AM

\* ARR1768 \*

PRODUCT => NALAUSF	
NET WEIGHT =>	0.126 KG
TARE =>	0.266 KG
GROSS WEIGHT =>	0.392 KG

2. Weighing the dissolution medium gal 111

1L of 0.025M NaLS = 1001.0 g

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001548

vessel	reset	amt weighed	signature	
1	yes	1001.6 g	[initials] 03/13/97	[initials] 3/13/97
2	yes	1001.6 g	[initials] 03/13/97	[initials] 3/13/97
3	yes	1001.6 g	[initials] 03/13/97	[initials] 3/13/97
4	yes	1001.6 g	[initials] 03/13/97	[initials] 3/13/97
5	yes	1001.6 g	[initials] 03/13/97	[initials] 3/13/97
6	yes	1001.6 g	[initials] 03/13/97	[initials] 3/13/97

### 3 - Weighing the tablets - gal 205

a 434 mg tablet contains 100 mg of fenofibrate

[See original for weight slip.]

### 4 - Dissolution conditions

Prolabo dissolutest gal 103 with paddles

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$       \      [initials] 03/13/97  
 speed = 75 rpm      /      [initials] 3/13/97      [initials] 03/11/97

5 ml of medium collected using a plastic 10ml syringe ref. Plastipack 302188 equipped with a Prolabo prefilter ref. 178398501.

Replacement of the collected medium with 5ml of new medium.

[initials] 07/03/97

5. Readings

[initials] 07/03/97

using KONTRON 922 930 spectrophotometer gal 108 in 2mm path length cuvette

filtration of samples through Millex HA 0.45µm filters ref Millipore SLHA025NB

TABLET PHARMA PASS 100MG LOT 361
----------------------------------

03/13/1996 3:48 PM

Lambda	No.	Value_E
--------	-----	---------

		AZ [unknown acronymn]
--	--	-----------------------

		air/air
--	--	---------

		NaLS/NaLS
--	--	-----------

		AZ [unknown acronymn]
--	--	-----------------------

		NaLS/NaLS
--	--	-----------

[See original for figures.]

[initials] [illegible date]

Highly confidential subject to protective order

FOURNIER 1001550

# DISSOLUTION

m:\commun\gingq\traidon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	100 mg fenofibrate tablet PHARMA PASS lot 361
DATE	03/13/97	NOTEBOOK NO.	LF 178ter p 64
INSTRUMENT	gal 103 108	FILE	m:\commun\gingq\donnbase\lf178ter\dissolution\lot 361
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	434	
theoretical dosage	100	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

\*without significant impact on processing the data

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001551

3/14/97

Dissolution of PHARMA PASS tablets  
with 100 mg fenofibrate  
lot 334

1. Preparation of the dissolution medium      0.025M NaLS

see p. 64

2. Weighing the dissolution medium    gal 111

1L of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	[initials] 03/14/97 [initials] 3/14/97
2	yes	1001.0 g	[initials] 03/14/97 [initials] 3/14/97
3	yes	1001.0 g	[initials] 03/14/97 [initials] 3/14/97
4	yes	1001.0 g	[initials] 03/14/97 [initials] 3/14/97
5	yes	1001.0 g	[initials] 03/14/97 [initials] 3/14/97
6	yes	1001.0 g	[initials] 03/14/97 [initials] 3/14/97

3. Weighing the tablets    gal 205

one 434 mg tablet contains 100 mg of fenofibrate

[initials] 07/03/97

[See original for weight slip.]

4. Dissolution conditions

Prolabo dissolutest with paddles gal 103

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$

[initials] 3/14/97 [initials] 03/14/97

speed = 75 rpm

[initials] 3/14/97 [initials] 03/14/97

5 ml of medium collected using a 10 ml syringe ref Plastipack 302188 equipped with a Prolabo prefilter ref 178 398 501.

The collected medium is replaced with 5 ml of new medium.

5. Readings

[initials] [07/03/97]

Using KONTRON 922 930 spectrophotometer gal 108 in 2mm path length cuvettes

Each sample is filtered prior to measurement through Millex HA 0.45 $\mu\text{m}$  ref Millipore SLHA025NB

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001553

TABLETS PHARMA PASS LOT 334 100MG FENOFIBRATE

03/14/1996 10:48 AM

Lambda No. Value\_E

AZ [unknown acronymn]  
yes/yes  
NaLS/NaLS  
AZ [unknown acronymn]  
NaLS/NaLS

- forgot to  
print T0

[See original for figures.]

[date and initials cut off]

Highly confidential subject to protective order

FOURNIER 1001554



## DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

### OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	100 mg fenofibrate tablet PHARMA PASS lot 334
DATE	03/14/97	NOTEBOOK NO.	LF 178ter p 68
INSTRUMENT	gal 103 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 334
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

### SAMPLE PREPARATION

theoretical mass	434	
theoretical dosage	100	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

[initials] 07/03/97

### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures. Dissolution form obscured by the following form - unobscured dissolution form appears on page 1001556.]

### DISSOLUTION OF PHARMA PASS TABLETS CONTAINING 100 MG OF FENOFIBRATE

time	lot 334	lot 351	lot 354	lot 358	lot 361
		[See original for figures.]			

[See original for graph.]

[initials] 07/03/97

Highly confidential subject to protective order.

FOURNIER 1001555

# DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	100 mg fenofibrate tablet PHARMA PASS lot 334
DATE	03/14/97	NOTEBOOK NO.	LF 178ter p 68
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 334
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	434	
theoretical dosage	100	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

[initials]  
07/03/97

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for figures.]

time

time

Highly confidential subject to protective order

FOURNIER 1001556

3/20/97

The dissolution medium chosen for the rest of the study is 0.025M NaLS

Dissolution of Canada gelcaps  
of 200mg lipidil micronized

Context

These dissolutions of CANADA gelcaps are for ~~the determination~~ the pharmacovigilance kinetic study.

2 lots are currently to be studied      lot 48  
lot 49

A. lot 48

1. Preparation of the 0.025M NaLS

molecular mass of NaLS = 288.4g

weighing the water:

tare 2.805 kg      [initials] 3/20/97    [initials] 03/20/97  
net: 20.230 - 2.805 = 17.425 kg  
gross: 20.230 kg      [initials] 3/20/97    [initials] 03/20/97

which is  $17.425 \times 0.025 \times 288.4 = 125.6\text{g}$  of NaLS to be weighed

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001557

# SIMPLE WEIGHING

Date weighed: 03/20/1997

11:21:05 AM

\* ARR1768 \*

PRODUCT => NALAUSF

NET WEIGHT => 0.126 KG

TARE => 0.262 KG

GROSS WEIGHT => 0.388 KG

## 2. Weighing the dissolution medium gal 111

1 liter of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	[initials] 03/20/97 [initials] 3/20/97
2	yes	1001.0 g	[initials] 03/20/97 [initials] 3/20/97
3	yes	1001.0 g	[initials] 03/20/97 [initials] 3/20/97
4	yes	1001.0 g	[initials] 03/20/97 [initials] 3/20/97
5	yes	1001.0 g	[initials] 03/20/97 [initials] 3/20/97
6	yes	1001.0 g	[initials] 03/20/97 [initials] 3/20/97

## 3. Dissolution conditions

Dissolutest with paddles gal 103

T° = 37°C±0.5°C [initials] 3/20/97 [initials] 03/20/97

speed = 75 rpm [initials] 3/20/97 [initials] 03/20/97

5 ml of medium collected at T: 5, 10, 15, 20, 30, 40, 50, 60, 120 min with a plastic 10 ml syringe ref Plastipack 302188 equipped with a Prolabo prefilter ref 178398501.

The collected medium is replaced with 5 ml of new medium.

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001558

#### 4. Measurements

Using KONTRON spectrophotometer 930 gal 108  
in 2mm path length cuvette  
filtration of samples through Millex HA 0.45µm ref SLHA025NB

GELCAPS LIP 200M CANADA LOT 48
--------------------------------

03/20/1996 3:47 PM

Lambda	No.	Value_E
--------	-----	---------

		AZ [unknown acronymn] air/air NaLS/NaLS
--	--	--

		AZ [unknown acronymn] NaLS/NaLS
--	--	---------------------------------

[See original for figures.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001559

# DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	lip200 M gelcaps Canada lot 48
DATE	03/20/97	NOTEBOOK NO.	Lf 178ter dissolution no.1 p72
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 48 Canada
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001560

3/21/97

B. Lot 49

1. Preparation of the dissolution medium 0.025M NaLS

see p72

2. Weighing the dissolution medium gal 111

1 L of NaLS = 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	[initials] 03/21/97 [initials] 03/21/97
2	yes	1001.0 g	[initials] 03/21/97 [initials] 03/21/97
3	yes	1001.0 g	[initials] 03/21/97 [initials] 03/21/97
4	yes	1001.0 g	[initials] 03/21/97 [initials] 03/21/97
5	yes	1001.0 g	[initials] 03/21/97 [initials] 03/21/97
6	yes	1001.0 g	[initials] 03/21/97 [initials] 03/21/97

3. Dissolution conditions

Prolabo dissolutest gal 103 with rotating paddles

T° = 37°C±0.5°C [initials] 3/21/97 [initials] 03/21/97  
speed = 75 rpm [initials] 3/21/97 [initials] 03/21/97

5 ml of medium collected at T = 5, 10, 15, 20, 30, 40, 50, 60, 120 min using a plastic 10 ml syringe ref Plastipack 302188 equipped with a Prolabo prefilter ref 178 398 501.  
The collected medium is replaced with 5 ml of new medium.

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001561

#### 4. Measurements

### DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

#### OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	gelcaps lip200 M Canada lot 49
DATE	03/21/97	NOTEBOOK NO.	lf 178ter dissolution no.1 p76
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 49 Canada
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

#### SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

#### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

#### RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

#### RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001562



#### 4. Measurements

Using KONTRON spectrophotometer 930 gal 108 in 2mm path length cuvettes

FIXED WAVELENGTH
------------------

03/21/1996 3:03 PM

Lambda	No.	Value_E
--------	-----	---------

		AZ [unknown acronymn] air/air NaLS/NaLS
--	--	--

		AZ [unknown acronymn] NaLS/NaLS
--	--	---------------------------------

[See original for figures.]

a drop fell into the [illegible]  
of the cuvette => reading redone

continuation of p 72 study

3/24/97

C. Lot 50

1. Preparation of the dissolution medium 0.025M NaLS

molecular mass of NaLS = 288.4g

weighing the water:

tare 2.803 kg [initials] 3/24/97 [initials] 03/24/97  
gross: 17.920 kg [initials] 3/24/97 [initials] 03/24/97  
net: 17.920 - 2.830 = 15.090 kg

which is  $15.090 \times 288.4 \times 0.025 = 108.8\text{g}$  of NaLS to be weighed

SIMPLE WEIGHING

Date weighed: 03/24/1997

10:44:12 AM

\* ARR1768 \*

PRODUCT => NALAUSF

NET WEIGHT => 0.109 KG

TARE => 0.265 KG

GROSS WEIGHT => 0.374 KG

2. Weighing the dissolution medium gal 111

1L of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signatures
1	yes	1001.0 g	[initials] 03/24/97 [initials] 3/24/97
2	yes	1001.0 g	[initials] 03/24/97 [initials] 3/24/97
3	yes	1001.0 g	[initials] 03/24/97 [initials] 3/24/97
4	yes	1001.0 g	[initials] 03/24/97 [initials] 3/24/97
5	yes	1001.0 g	[initials] 03/24/97 [initials] 3/24/97
6	yes	1001.0 g	[initials] 03/24/97 [initials] 3/24/97

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001564

### 3. Dissolution conditions

Prolabo dissolutest gal 103 equipped with rotating paddles

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$

[initials] 3/24/97 [initials] 03/24/97

speed = 75 rpm

[initials] 3/24/97 [initials] 03/24/97

5 ml sample of medium collected at T = 5, 10, 15, 20, 30, 40, 50, 60, 120 min using a plastic 10 ml syringe ref Plastipack 302188 equipped with a Prolabo prefilter ref 178398501.

The collected medium is replaced with 5 ml of new medium.

### 4. Measurements

Using KONTRON spectrophotometer 930 gal 108

in 2mm path length cuvettes

filtration of samples through Millex HA 0.45  $\mu\text{m}$  filters ref Millipore SLHA025NB.

FIXED WAVELENGTH
------------------

03/24/1996 3:24 PM

Lambda	No.	Value_E
--------	-----	---------

		AZ [unknown acronymn] air/air
--	--	-------------------------------

		NaLS/NaLS
--	--	-----------

		AZ [unknown acronymn] NaLS/NaLS
--	--	---------------------------------

[See original for figures.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001565

# DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	gelcaps lip200 M Canada lot 50
DATE	03/24/97	NOTEBOOK NO.	If 178ter dissolution no.1 p78
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 50 Canada
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

mass of test sample  
quantity of active substance

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001566

3/25/97

Dissolution of lipanthyl 200 M  
lot 2177

Context: compare this dissolution with those for the lipanthyl 200 gelcaps - CANADA lots.

1. Preparation of the 0.025M NaLS

see p78.

2. Weighing the dissolution medium gal 111

1L of 0.025M NaLS = 1001.0g

vessel	reset	amt weighed	signatures
1	yes	1001.0 g	[initials] 03/25/97 [initials] 3/25/97
2	yes	1001.0 g	[initials] 03/25/97 [initials] 3/25/97
3	yes	1001.0 g	[initials] 03/25/97 [initials] 3/25/97
4	yes	1001.0 g	[initials] 03/25/97 [initials] 3/25/97
5	yes	1001.0 g	[initials] 03/25/97 [initials] 3/25/97
6	yes	1001.0 g	[initials] 03/25/97 [initials] 3/25/97

3. Operating conditions

dissolutest with paddles gal 103

T° = 37°C±0.5°C  
speed = 75 rpm

[initials] 3/25/97 [initials] 03/25/97  
[initials] 3/25/97 [initials] 03/25/97

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001567

5 ml of medium collected at T = 5, 10, 15, 20, 30, 40, 50, 60, 120 min using a plastic 10 ml syringe ref Plastipack 302188 equipped with a Prolabo prefilter ref 178398501.

The collected ~~new~~ medium is replaced with 5 ml of new medium.

#### 4. Measurements

KONTRON 930 spectrophotometer gal 108  
in 2mm path length cuvette  
filtration of samples through Millex HA 0.45µm ref Millipore SLHA025NB

GELCAPS LIP200M LOT 2177
--------------------------

03/25/1996 11:22 AM

Lambda	No.	Value_E
--------	-----	---------

		AZ [unknown acronymn]
		air/air
		NaLS/NaLS
		AZ [unknown acronymn]
		NaLS/NaLS

[See original for figures.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001568

# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	gelcaps lip200 M lot 2177
DATE	03/25/97	NOTEBOOK NO.	If 178ter dissolution no.1 p81
INSTRUMENT	gal 103 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2177
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001569

		Canada lots		
time	lot 2177	lot 48	lot 49	lot 50
		[See original for figures.]		

source data lab notebook  
lf178ter dissolution no. 1  
pages 72-83

### comparison of dissolutions of lip200M gelcaps

[See original for graph.]

time in minutes

Lot 2177 is not different from the CANADIAN lots, but note the difference at 10 min, 15 min and 20 min of approximately 10%.

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001570



4/3/97

Dissolution of lot 2394/01 RG

two hardnesses were tested = 14 and 18 kg

A. Hardness of 14 kg

1. Preparation of the 0.025M NaLS

molecular mass of NaLS = 288.4 g

Weighing the water:

tare 3.080 kg [initials] 4/3/97 [initials] 04/03/97  
gross: 20.870 kg [initials] 4/3/97 [initials] 04/03/97  
net: 20.870 - 3.080 = 17.790 kg

which is  $17.790 \times 0.025 \times 288.4 = 128.3\text{g}$

SIMPLE WEIGHING

Date weighed: 04/03/1997

11:26:55 AM

\* ARR1768 \*

PRODUCT => NALAUSF

NET WEIGHT => 0.128 KG

TARE => 0.154 KG

GROSS WEIGHT => 0.282 KG

2. Weighing the dissolution medium gal 111

[initials] 4/4/97

1L of 0.025M NaLS = 1001.0 g

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001571

vessel	reset	amt weighed	signatures	
1	yes	1001.0 g	[initials] 04/03/97	[initials] 4/3/97
2	yes	1001.0 g	[initials] 04/03/97	[initials] 4/3/97
3	yes	1001.0 g	[initials] 04/03/97	[initials] 4/3/97
4	yes	1001.0 g	[initials] 04/03/97	[initials] 4/3/97
5	yes	1001.0 g	[initials] 04/03/97	[initials] 4/3/97
6	yes	1001.0 g	[initials] 04/03/97	[initials] 4/3/97

3. Weighing the tablets gal 205

One 694.4 mg tablet contains 160 mg of fenofibrate

[See original for weight slip.]

[initials] 4/4/97

4. Dissolution conditions

Prolabo dissolutest equipped with rotating paddles gal 103

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$   
speed = 120 rpm

[initials] 4/3/97 [initials] 04/03/97  
[initials] 4/3/97 [initials] 04/03/97

5 ml sample of medium collected at T = 5, 10, 15, 20, 30, 40, 50, 60, 120 min

[initials] 07/03/97

using a plastic 10 ml syringe ref [text cut off] equipped with a Prolabo prefilter ref  
178398501.

The collected medium is replaced with 5 ml of new medium.

Dissolution to be redone. p.90

speed 120 rpm  
instead of 75 rpm

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001573

[This page is blank and crossed out.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001574

4/4/97

B. Hardness of 18 kg

694 Preparation of the 0.025M NaLS

see p85

2. Weighing the dissolution medium gal 111

1L of 0.025M NaLS = 1001.0g

vessel	reset	amt weighed	signatures	
1	yes	1001.0 g	[initials] 04/04/97	[initials] 4/4/97
2	yes	1001.0 g	[initials] 04/04/97	[initials] 4/4/97
3	yes	1001.0 g	[initials] 04/04/97	[initials] 4/4/97
4	yes	1001.0 g	[initials] 04/04/97	[initials] 4/4/97
5	yes	1001.0 g	[initials] 04/04/97	[initials] 4/4/97
6	yes	1001.0 g	[initials] 04/04/97	[initials] 4/4/97

3. Weighing the tablets gal 205

One tablet of 694.4 mg contains 160 mg of fenofibrate

[See original for weight slip.]

694 Operating conditions

Prolabo dissolutest gal 103 equipped with rotating paddles

T° = 37°C±0.5°C [initials] 04/04/97

speed = 120 rpm [initials] 04/04/97

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001575

Hardness – 14 kg  
continued from page 85

2. Weighing the dissolution medium gal 111  
1L of 0.025M NaLS = 1001.0g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	[initials] 04/04/97 [initials] 4/4/97
2	yes	1001.0 g	[initials] 04/04/97 [initials] 4/4/97
3	yes	N/A	[initials] 04/04/97 [initials] 4/4/97
4	yes	1001.0 g	[initials] 04/04/97 [initials] 4/4/97
5	yes	1001.0 g	[initials] 04/04/97 [initials] 4/4/97
6	yes	1001.0 g	[initials] 04/04/97 [initials] 4/4/97

Not enough 0.025M NaLS dissolution in [illegible – probably 5 lots] vessels

3. Weighing the tablets gal 205

A 694.4 mg tablet contains 160 mg of fenofibrate

[See original for weight slip.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001576

#### 4. Dissolution conditions

##### DISSOLUTION

m:\commun\ging\traitdon\distern5  
date printed: 02/06/97

##### OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	160 mg fenofibrate tablet lot 2394/01 Rg 14 kg hardness
DATE	04/04/97	NOTEBOOK NO.	LF 178ter p 90
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donbase\lf178ter\dissolution\01Rg 14 kg
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

##### SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

##### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

##### RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

##### RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001577

#### 4. Dissolution conditions

Prolabo dissolutest gal 103 equipped with rotating paddles

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$

[initials] 4/4/97 [initials] 04/04/97

speed = 75 rpm

[initials] 4/4/97 [initials] 04/04/97

5 ml sample of medium collected at T = 5, 10, 15, 20, 30, 40, 50, 60 min using a plastic 10 ml syringe ref Plastipack 302188 equipped with a Prolabo prefilter ref 178398501. The collected medium is replaced with 5 ml of new medium.

#### 4. Measurements

KONTRON spectrophotometer 930 gal 108

in 2mm path length cuvette

filtration of samples through Millex HA filters ref Millipore SLHA025NB.

FIXED WAVELENGTH
------------------

04/04/1996 10:51 AM

Lambda	No.	Value_E
--------	-----	---------

[See original for figures.]



4/7/97

Dissolution of tablets lf178  
(lot 348 Canada)  
coated with OPADRY OY

lot 2393/01 RG

1. Preparation of 0.025M NaLS

([illegible] not working, removed manually)

molecular mass of NaLS = 288.4 g

tare water = 2.725 kg [initials] 4/7/97 [initials] 04/07/97  
gross water = 28.830 kg [initials] 4/7/97 [initials] 04/07/97  
net = 28.830 - 2.725 = 26.105 kg

which is  $26.105 \times 288.4 \times 0.025 = 188.2$  g of NaLS

gal 011 tare = 635.7 g [initials] 4/7/97 [initials] 04/07/97  
gross = 824.0 g [initials] 4/7/97 [initials] 04/07/97  
net = 824.0 - 635.7 = 188.3g

2. Weighing the dissolution medium gal 111

1L of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	04/07/97 [initials] [initials] 4/7/97
2	yes	1001.0 g	04/07/97 [initials] [initials] 4/7/97
3	yes	1001.0 g	04/07/97 [initials] [initials] 4/7/97
4	yes	1001.0 g	04/07/97 [initials] [initials] 4/7/97
5	yes	1001.0 g	04/07/97 [initials] [initials] 4/7/97
6	yes	1001.0 g	04/07/97 [initials] [initials] 4/7/97

[initials] 06/02/97

[initials] 07/03/97

3. Weighing the tablets gal 205

the tablets contain 100 mg of fenofibrate

[See original for weight slip.]

4. Dissolution conditions

Prolabo dissolutest gal 103 equipped with rotating paddles

$T^{\circ} = 37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  [initials] 4/7/97 [initials] 04/07/97  
speed = 75 rpm [initials] 4/7/97 [initials] 04/07/97

5 ml sample of medium collected at T = 5, 10, 15, 20, 30, 40, 50, 60 min using a plastic  
10 ml syringe ref Plastipack 302188 equipped with a Prolabo prefilter ref 178398501.  
The collected medium is replaced with 5 ml of new medium.

4. Readings

KONTRON spectrophotometer 930 gal 108  
2mm path length cuvettes

LF178TER LOT 2393/01RG COATED OPADRY OY

04/07/1996 1:[text cut off] PM

	Lambda	No.	Value_E
[initials] 06/02/97			AZ [unknown acronymn] air/air
			NaLS/NaLS
			AZ [unknown acronymn] NaLS/NaLS

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001580

# DISSOLUTION

m:\commun\ginq\traitdon\distern5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	tablets coated with OPADRY OY lot 2393/01RG
DATE	04/07/97	NOTEBOOK NO.	LF 178ter dissolutionpage 92
INSTRUMENT	gal 103 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2393rg01
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

## SAMPLE PREPARATION

[initials] 06/02/97

theoretical mass	100	
theoretical dosage	100	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001581

[See original for figures.]

04/07/97

LOT RG 2394/01  
hardness - 14 kg

1. Preparation of dissolution medium  
see page 92

2. Weighing the dissolution medium Balance GAL 111  
1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025 NaLS	verifiers
1	yes	1001.0 g	[initials] 4/7/97 [initials] 04/07/97
2	yes	1001.0 g	[initials] 4/7/97 [initials] 04/07/97
3	yes	1001.0 g	[initials] 4/7/97 [initials] 04/07/97
4	yes	1001.0 g	[initials] 4/7/97 [initials] 04/07/97
5	yes	1001.0 g	[initials] 4/7/97 [initials] 04/07/97
6	yes	1001.0 g	[initials] 4/7/97 [initials] 04/07/97

Dissolution conditions

Dissolutest	T° = 37°C±0.5	[initials] 4/7/97 [initials] 04/07/97
GAL 091	speed = 75 rpm	[initials] 4/7/97 [initials] 04/07/97

3. Weighing the tablets Balance GAL ~~214~~ 205 [initials] 04/07/97

no. 3 and 5: reset not exact. Error taken into account for the net value entered on the calculation sheet. [initials] 07/11/97

[See original for weight slip.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001583

4. Readings

UV GAL 233 290 nm cell 2mm chrono GAL123

LF 178 TER LOT RG 2394/01 14 KG

04/06/1997 10:53 PM

Lambda No. Value\_E

[text obscured]

due to a problem

[illegible - probably with cleanliness of] the cell during  
the reading...?

[initials, date illegible]

1) AZ [unknown acronym] Air/Air

2) NaLS/NaLS

3

290.0 30 1.2406 1 290.0 60 1.3949\_1

**DISSOLUTION**

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D. LECRIT	TITLE	LF 178 TER RG 2394/01 at 14 KG
DATE	04/07/97	NOTEBOOK NO.	178 TER no. 1 p 96 95
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2394RG01 14 kg
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

wrong masses for cell 3 and 5 see page 95 [initials] 07/11/97

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

DISSOLUTION READINGS

[initials] 07/11/97

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

abnormal value: 1.115, not taken into account [initials] 04/07/97

RESULTS — % DISSOLVED

[See original for figures.]

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001585

LOT RG 2394/01  
Hardness - 18 kg

1. Preparation of dissolution medium  
see page 92

2. Weighing the dissolution medium Balance GAL 111  
1 liter 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifications
1	yes	1001.0 g	[initials] 04/07/97 [initials] 04/07/97
2	yes	1001.0 g	[initials] 04/07/97 [initials] 04/07/97
3	yes	1001.0 g	[initials] 04/07/97 [initials] 04/07/97
4	yes	1001.0 g	[initials] 04/07/97 [initials] 04/07/97
5	yes	1001.0 g	[initials] 04/07/97 [initials] 04/07/97
6	yes	1001.0 g	[initials] 04/07/97 [initials] 04/07/97

Dissolution conditions

Dissolutest                      T° = 37°C±0.5                      [initials] 4/7/97 [initials] 04/07/97  
GAL 091                              speed = 75 rpm                      [initials] 4/7/97 [initials] 04/07/97

3. Weighing the tablets Balance GAL 214 205 [initials] 04/07/97

[See original for weight slip.]

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001586



UV GAL 255 290 nm 2mm cell

LF 178 TER LOT RG 2394/01 18 KG

04/07/1997 2:23 AM

Lambda No. Value\_E

1 AZ [unknown acronym] Air/Air

2 NaLS/NaLS

[See original for figures.]

[text obscured]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001587

290.0 61 1.3210\_1  
290.0 62 1.3349\_1

**DISSOLUTION**

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR D. LECRIT TITLE LF 178 TER RG 2394/01 at 18 KG  
DATE 04/07/97 NOTEBOOK NO. LF 178 TER no. p98 97  
INSTRUMENT GAL 233 FILE m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2394RG01 18 kg  
GAL 091  
WAVELENGTH 290 cm ELUANT 0.025M NaLS  
CUVETTE in mm 2 mm STIRRING 75 rpm

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

[initials] 07/03/97

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001588

UV GAL 233 290 nm 2mm cell

LF 178 TER LOT RG 2394/01 18 KG

04/07/1997 2:23 AM

Lambda No. Value\_E

- 1 AZ [unknown acronym] Air/Air
- 2 NaLS/NaLS
- 3 AZ [unknown acronym] NaLS/NaLS

[See original for figures.]

\*abnormal value  
(% dissolved < previous T)  
not retained  
\* problem with reading  
new measurement done  
on the same sample  
[initials] 06/07/97

DISSOLUTION

m:\commun\ging\traidon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D. LECRIT	TITLE	LF 178 TER RG 2394/01 at 18 KG
DATE	04/07/97	NOTEBOOK NO.	LF 178 TER no. p98 97
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2394RG01 18 kg
WAVELENGTH	290 cm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
quantity of active substance						

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

[initials] 07/03/97

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[See original for figures.]

Highly confidential subject to protective order

FOURNIER 1001589

4/7/97

Assay of fenofibrate in  
tablets of If178 ter  
lot 2394/01 RG at 14 kg hardness  
at 18 kg hardness

Assay done by HPLC according to procedure 02DAP004.06/05.

"Identification and assay of fenofibrate in 200 mg gelcaps of micronized  
fenofibrate"

Chromatographic conditions

- HPLC gal 087.089.261
- column RP 18 (250x4) 5µm (column no. 2) lichrosorb Merck ref 50333
- ACN/H<sub>2</sub>O 70/30 ACN ref Merck 14291  
acidified to pH = 2.5
- orthophosphoric acid Prolabo ref 2624.295
- tetrahydrofurane RS ref 28-556-293. Prolabo.
- flow rate: 1.2 ml/min
- T° = 35°C
- injection: loop of 10 µl (the loop is rinsed after injection with [text cut off])
- wavelength: 286 nm
- analysis time: 10 min for the STAR  
11 min for the LC 5000  
12 min for the 9090

[initials] [illegible date]

Highly confidential subject to protective order

FOURNIER 1001590

1. Preparation of control solutions gal 205

1.1. Stock solutions for controls: (do twice)

Introduce about 25.0 mg of accurately weighed fenofibrate arr1241 into a 50 ml volumetric flask.

Dissolve the feno in 5.0 ml of THF in a magnetic stirrer.

Qsp with ACN.

Magnetic stirring for 30 minutes

[See original for weight slip.]

= stock control 1 and stock control 2

tare

tare

net

net

gross

gross

1.2 Working solutions for controls

Using a pipette, introduce 2 ml of the stock solution (control) into a 20 ml volumetric flask.

Qsp with mobile phase then homogenize.

T1 at 0.514  $\mu\text{g}/10\ \mu\text{l}$

T2 at 0.512  $\mu\text{g}/10\ \mu\text{l}$

2. Preparation of test solutions

2.1 Stock solutions for tests

to be done twice

carefully crush 10 tablets in a mortar. Mix.

Weigh about 350.0 mg, accurately weighed, of mixture in 150 ml flask.

[initials] 07/03/[text cut off]

[See original for weight slip.]

[brut = gross]

$$20 \quad 250.1 + 101.3 = 351.4 \text{ mg}$$

Add 15.0 ml of THF and 7.5 ml of mobile phase

Magnetic stirring for 45 min

Qsp with ACN

Stir for 45 min

### 2.2. Working solutions for tests

Dilute each stock working solution (for tests) to 1/25.

Qsp with mobile phase.

### 3. Injections and results

The chromatograms are in appendix 2.

\* inject 5 times T1 and calculate the calibration factor F.

$$F = \frac{CF}{AF} \text{ <---- T1 feno concentration in } \mu\text{g}/10\mu\text{l}$$

AF <----- peak area

area	F
[See original for figures.]	

[initials] 07/03/97  
[equation] for T1

[initials] 07/03/97  
[equation] satisfactory

mean of last 3 tests

[equation]

[initials] 07/03/97

calculating the calibration factor for T2

[See original for figures.]

\* verification of controls

$$\frac{T_2}{T_1} \times 100 = \frac{1.455 \times 10^{-6}}{1.464 \times 10^{-6}} = 99.4\% \text{ controls ok accepted}$$

\* mean of calibration factors =

$$MF = \frac{1.455 \times 10^{-6} + 1.464 \times 10^{-6}}{2} = 1.460 \times 10^{-6}$$

\* dose of fenofibrate

[text crossed out]

in each preparation (see procedure for expanded formula)

$$375 \times \frac{MF \times A1 \times MM}{PE} \quad 694.4\text{mg}$$

for 14 kg A

$$375 \times \frac{1.460 \times 10^{-6} \times 144161 \times 394.4}{351.4} =$$

[See original for formulas.]

for

for

tablet

for

tablet

for

tablet

No injection of 18 kg B

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001593

whether they have a hardness of 18 kg or 14 kg, the tablets come from the same mixture and one can therefore take the mean of these results:

$$\frac{155.97 + 192.05 + 152.74}{3} = 166.92 \text{ mg/tablet.}$$

lot 2394/01RG or 167 mg/tablet.

note the large value of 192.05 mg.

The corresponding chromatograms are saved in appendix 2.

[initials] 07/03/97



LOT RG 2396/01  
hardness - 18 kg

1. Preparation of the dissolution medium  
See page 92

2. Weighing the dissolution medium Bal GAL 111  
1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.1 g	[initials] 4/8/97 [initials] 04/08/97
2	yes	1001.1 g	[initials] 4/8/97 [initials] 04/08/97
3	yes	1001.0 g	[initials] 4/8/97 [initials] 04/08/97
4	yes	1001.0 g	[initials] 4/8/97 [initials] 04/08/97
5	yes	1001.1 g	[initials] 4/8/97 [initials] 04/08/97
6	yes	1001.1 g	[initials] 4/8/97 [initials] 04/08/97

Conditions

Dissolutest	T° 37°C±0.5	[initials] 4/8/97 [initials] 04/08/97
GAL 091	speed 75 rpm	[initials] 4/8/97 [initials] 04/08/97

3. Weighing the tablets BAL GAL 205

[See original for weight slip.]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001595

4. Readings

UV GAL 233 290 nm 2mm cell Chrono GAL 123

LF 178 TER LOT RG 2396/01 18 KG

4/7/1997 10:57 PM

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

[initials and date obscured]

Highly confidential subject to protective order

FOURNIER 1001596

#### 4. Readings

UV GAL 233 290 nm 2mm cell Chrono GAL 123

LF 178 TER LOT RG 2396/01 18 KG

4/7/1997 10:57 PM

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

#### DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

#### OPERATING CONDITIONS AND SETTINGS

OPERATOR	D LECRIT	TITLE	LF 178 TER RG 2396/01 at 18 KG
DATE	04/08/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 105
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbases\lf178ter\dissolution\lot 2396RG01 18 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

#### SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

#### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001597

290.0 30 1.1769\_1 290.0 60 1.390\_1

[initials]

**DISSOLUTION**

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2396/01 at 18 KG
DATE	04/08/97	NOTEBOOK NO.	LF 178 TER no. 1 p 105
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2396RG01 18 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

DISSOLUTION READINGS

[initials] 07/03/97

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001598

4/8/97

Dissolution of tablets  
coated with OPADRY OY  
lot 2393/01RG stored 15 days at 75% RH

1. Preparation of the 0.025M NaLS

molecular mass of NaLS = 288.4 g

weighing the water: tare - 2.800 kg [initials] 4/8/97 [initials] 04/08/97  
gross = 22.590 kg [initials] 4/8/97 [initials] 04/08/97  
net = 22.590 - 2.800 = 19.790 kg

which is  $19.790 \times 288.4 \times 0.025 = 143$  g of NaLS to be weighed out

tare = 161.6 g [initials] 4/8/97 [initials] 04/08/97  
gross = 304.6 g [initials] 4/8/97 [initials] 04/08/97  
net = 143.0 g

2. Weighing the dissolution medium gal 111

1 liter of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	[initials] 04/08/97 [initials] 4/8/97
2	yes	1001.0 g	[initials] 04/08/97 [initials] 4/8/97
3	yes	1001.0 g	[initials] 04/08/97 [initials] 4/8/97
4	yes	1001.0 g	[initials] 04/08/97 [initials] 4/8/97
5	yes	1001.0 g	[initials] 04/08/97 [initials] 4/8/97
6	yes	1001.0 g	[initials] 04/08/97 [initials] 4/8/97

[initials] 06/02/97  
[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001599

3. Weighing the tablets gal 205

[See original for weight slip.]

4. Dissolution conditions

Prolabo dissolutest gal 103 equipped with rotating paddles

T° = 37°C±0.5 [initials] 04/08/97 [initials] 4/8/97

speed = 75 rpm [initials] 04/08/97 [initials] 4/8/97

5. Readings

KONTRON 930 spectrophotometer gal 108

2mm path length cuvettes

LF 178TER COATED OPADRY OY 15 DAYS AT 75 HR
---

04/08/1996 1:40 PM

Lambda No. Value\_E

[See original for figures.]

[initials] 06/02/97

[initials] [illegible date]

Highly confidential subject to protective order

FOURNIER 1001600

# DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	tablets coated OPADRY OY lot 2393/01RG 15 days at 75% RH
DATE	04/08/97	NOTEBOOK NO.	LF 178ter dissolution page 106
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2393rg01 75rh
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2mm	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	100	
theoretical dosage	100	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

[initials] 06/02/97

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001601

04/06/97

LOT RG 2396/01  
Hardness - 14 kg

1. Preparation of dissolution medium  
see page 106

2. Weighing the dissolution medium Bal GAL 111  
1 liter of 0.025M NaLS → 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.0 g	[initials] 4/8/97 [initials] 04/08/97
2	yes	1001.1 g	[initials] 4/8/97 [initials] 04/08/97
3	yes	1001.0 g	[initials] 4/8/97 [initials] 04/08/97
4	yes	1001.0 g	[initials] 4/8/97 [initials] 04/08/97
5	yes	1001.0 g	[initials] 4/8/97 [initials] 04/08/97
6	yes	1001.1 g	[initials] 4/8/97 [initials] 04/08/97

Conditions

Dissolutest	T° 37°C±0.5	[initials] 04/08/97 [initials] 4/10/97
GAL 091	speed 75 rpm	[initials] 04/08/97 [initials] 4/10/97

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

[initials] 07/03/97 [text cut off]

Highly confidential subject to protective order

FOURNIER 1001602



4. Readings

UV GAL 233 290nm cell 2mm chrono GAL 123

LF 178 TER LOT RG2396/01 14 KG
--------------------------------

[See original for figures.]

Lambda No. Value\_E

AZ [expansion unknown] Air/Air

[text obscured]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001603

# DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2396/01 at 14 KG
DATE	04/08/97	NOTEBOOK NO.	178 TER no. 1 p 110
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2396RG01 14 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

[initials] 07/03/97

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001604

4. Readings

UV GAL 233 290 nm 2mm cell Chrono GAL 123

LF 178 TER LOT RG 2396/01 14 KG

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

error with  
centrifuge  
[initials] 04/08/97

**DISSOLUTION**

m:\commun\gingq\traitdon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D LECRIT	TITLE	LF 178 TER RG 2396/01 at 14 KG
DATE	04/08/97	NOTEBOOK NO.	178 TER no. 1 p. 110
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\gingq\donnbase\lf178ter\dissolution\lot 2396RG01 14 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[cut off]

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001605

Assay of fenofibrate in  
mixtures prior to compression  
lot 2394/01 RG  
lot 2396/01 RG

Assay done by HPLC as per procedure 02DAP004-06/05

Chromatographic conditions  
see p. 99

1. Preparation of the control solutions

balance DAP no. 5

1.1 Stock solutions for controls: (do twice)

Introduce about 25.00 mg of accurately weighed fenofibrate arr1241 into a 50 ml volumetric flask.

\*\*\*\* 04/08/97 08:56 AM \*\*\*

\*\*\*\* 04/08/97 08:59 AM \*\*\*\*

=====

Sample: FENOFIBRATE T1

=====

Sample: FENOFIBRATE T2

Control No.: ARR 1241

Control No.: ARR 1241

Operator: AG

Operator: AG

Tare weight: 37073.13 g

Tare weight: 37365.84 g

Gross weight: 37098.52 g

Gross weight: 37393.42 g

NET WEIGHT: 25.39 g

NET WEIGHT: 27.58 g

! The Epson prints g although I weighed in mg.

Add 5 ml of THF

Qsp with ACN.

Magnetic stirring for 30 minutes.

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001606

yielding TM1 and TM2

1.2 Working solutions for controls

Dilute to 2:20 in the mobile phase

yielding:

T1 at 0.5078 µg/10 µl

T2 at 0.5516 µg/10 µl

2. Preparation of test solutions balance Dap 3

2.1 Stock solutions for tests

to be done twice

Accurately weigh about 350 mg of mixture in 150 ml flask.

\*\*\*\* 04/08/97 09:28 AM \*\*\*

=====

Sample: LF 178TER  
Control No.: 2394RG01 1  
Operator: AG

Tare weight: 19456.64 g

Gross weight: 19805.45 g

NET WEIGHT: 348.81 g

\*\*\*\* 04/08/97 09:31 AM \*\*\*\*

=====

Sample: LF 178TER  
Control No.: 2394RG01 2  
Operator: AG

Tare weight: 18884.78 g

Gross weight: 19236.38 g

NET WEIGHT: 351.60 g

\*\*\*\* 04/08/97 09:38 AM \*\*\*

=====

Sample: LF 178TER  
Control No.: 2396RG01  
Operator: AG 4

Tare weight: 19344.91 g

Gross weight: 19695.50 g

NET WEIGHT: 350.59 g

\*\*\*\* 04/08/97 09:34 AM \*\*\*\*

=====

Sample: LF 178TER  
Control No.: 2396RG01 3  
Operator: AG

Tare weight: 20139.44 g

Gross weight: 20486.63 g

NET WEIGHT: 347.19 g

! The Epson prints g although I weighed in mg.

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001607

Add 15 ml of THF + 7.5 l of mobile phase.

Magnetic stirring for 45 min

Qsp with ACN.

Magnetic stirring for 45 min.

## 2.2. Working solutions for tests

Dilute each stock working solution (for tests) to 1:25.

Qsp with mobile phase.

## 3. Injections and results

The chromatograms are in appendix 2.

\* inject T1 5 times and calculate the calibration factor F.

area  [See original for figures.]	$F = \frac{CF}{area}$ <---- feno concentration in $\mu g/10\mu l$	[See original for equations.]  mean of last 3 injections
--	---	--

\* calculating the calibration factor for T2

Area

F

[See original for figures. and equation]

[initials] 07/03/97

\* verification of controls

$$\frac{F_{T2}}{F_{T1}} \times 100 = \frac{1.497 \times 10^{-6} \times 100}{1.468 \times 10^{-6}} = 102.0\% \quad \text{controls accepted}$$

\* mean of calibration factors

$$MF = \frac{F_{T1} + F_{T2}}{2} = \frac{1.497 \times 10^{-6} + 1.468 \times 10^{-6}}{2}$$
$$MF = 1.483 \times 10^{-6}$$

\* dosage of fenofibrate per tablet

see procedure for expanded formula

$$\frac{375 \times MF \times \text{Area} \times 694.4}{PE}$$

[See original for formulas.]

[initials] [illegible date]

lot 2394/01 RG = 155 mg of fenofibrate/tablet

lot 2396/01 RG = 152 mg of fenofibrate/tablet

[initials] 07/03/97



LOT RG 2397/01  
hardness - 14 kg

1. Preparation of the dissolution medium

See page 106

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers	
1	yes	1001.0 g	[initials] 04/09/97	[initials] 04/09/97
2	yes	1001.1 g	[initials] 04/09/97	[initials] 04/09/97
3	yes	1001.0 g	[initials] 04/09/97	[initials] 04/09/97
4	yes	1001.0 g	[initials] 04/09/97	[initials] 04/09/97
5	yes	1001.0 g	[initials] 04/09/97	[initials] 04/09/97
6	yes	1001.0 g	[initials] 04/09/97	[initials] 04/09/97

Conditions

Dissolutest	T° 37°C±0.5	[initials] 04/09/97	[initials] 04/09/97
GAL 091	speed 75 rpm	[initials] 04/09/97	[initials] 04/09/97

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

[initials] 07/03/97

4. Readings

Spectro GAL 233

Chronometer GAL 123

LF 178 TER LOT RG 2397/01 14 KG

4/9/1997 12:28 PM

Lambda	No.	Value_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

[initials and date obscured]

Highly confidential subject to protective order

FOURNIER 1001612

#### 4. Readings

Spectro GAL 233

Chronometer GAL 123

LF 178 TER LOT RG 2397/01 14 KG

4/9/1997 12:28 PM

Lambda No. Value\_E  
Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

#### DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

#### OPERATING CONDITIONS AND SETTINGS

OPERATOR	D LECRIT	TITLE	LF 178 TER RG 2397/01 at 14 KG
DATE	04/09/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 117
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2397RG01 14 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

#### SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

#### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001613

290.0 30 1.2297\_1 290.0 60 1.4057\_7

**DISSOLUTION**

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2397/01 at 14 KG
DATE	04/09/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 117
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2397RG01 14 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

DISSOLUTION READINGS

Control 100 mg/l

volume sampled in ml

[initials] 07/03/97

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001614

LOT RG 2397/01  
hardness - 14 18 kg  
[initials]

1. Preparation of the dissolution medium

- weighing the water Bal GAL 014

tare = 2.730 kg [initials] 04/10/97 [initials] 4/10/97  
gross = 25.080 kg [initials] 04/10/97 [initials] 4/10/97  
net = 25.080 - 2.730 = 22.350

for a medium of 0.025M (MW 288.4g)

mass of NaLS = 22.350 x 288.4 x 0.025 = 161.1 g

- weighing the NaLS Bal GAL 011

tare = 153.5 g then reset [initials] 04/10/97 [initials] 4/10/97  
net = 161.1 g [initials] 04/10/97 [initials] 4/10/97 [initials] 04/10/97

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.1 g	[initials] 04/10/97 [initials] 04/10/97
2	yes	1001.1 g	[initials] 04/10/97 [initials] 04/10/97
3	yes	1001.0 g	[initials] 04/10/97 [initials] 04/10/97
4	yes	1001.0 g	[initials] 04/10/97 [initials] 04/10/97
5	yes	1001.0 g	[initials] 04/10/97 [initials] 04/10/97
6	yes	1001.0 g	[initials] 04/10/97 [initials] 04/10/97

Conditions

Dissolutest  
GAL 091

T° = 37°C±0.5  
speed = 75 rpm

[initials] 4/10/97 [initials] 04/10/97  
[initials] 4/10/97 [initials] 04/10/97

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001615

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

4. Readings

Spectro GAL 233

Chronometer GAL 123

LF 178 TER LOT RG 2397/01 18 KG

4/10/1997 12:28 PM

Lambda	No.	Value_E
--------	-----	---------

Lambda	No.	Value_E
--------	-----	---------

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001616

# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2397/01 at 18 KG
DATE	04/10/97	NOTEBOOK NO.	LF 178 TER no. 1 p 120
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2397RG01 18 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001617

4/10/97

Dissolution of LF178ter tablets  
Lot 2398/01RG hardness of 14 kg

1. Preparation of the 0.025M NaLS

see p. 118

2. Weighing the dissolution medium gal 111

1 liter of 0.025M NaLS = 1001.0 g

vessel	reset	amt weighed	signature
1	yes	1001.0 g	[initials] 04/10/97 [initials] 4/10/97
2	yes	1001.0 g	[initials] 04/10/97 [initials] 4/10/97
3	yes	1001.0 g	[initials] 04/10/97 [initials] 4/10/97
4	yes	1001.0 g	[initials] 04/10/97 [initials] 4/10/97
5	yes	1001.0 g	[initials] 04/10/97 [initials] 4/10/97
6	yes	1001.0 g	[initials] 04/10/97 [initials] 4/10/97

3. Weighing the tablets gal 205

1 tablet of 694.4 mg contains 160 mg of fenofibrate.

[See original for weight slip.]

[initials] 07/09[text cut off]

Highly confidential subject to protective order

FOURNIER 1001618



4. Operating conditions

Prolabo dissolutest gal 103 equipped with rotating paddles

T° = 37°C±0.5°C      [initials] 4/10/97    [initials] 04/10/97  
speed: 75 rpm            [initials] 4/10/97    [initials] 04/10/97

5. Readings

using KONTRON 930 gal 103  
2 mm path length cuvettes  
filtration through Millex HA25 ref SLHA025NB Millipore

LF 178TER TABLETS LOT 2398/01RG 14 KG
---------------------------------------

04/10/1996 1:18 PM

Lambda	No.	Value_E
--------	-----	---------

	AZ [expansion unknown] air/air	
	NaLS/NaLS	
	AZ [expansion unknown] NaLS/NaLS	

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001619

# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	LF 178 ter 2398/01RG at 14 KG
DATE	04/10/97	NOTEBOOK NO.	178 TER no. 1 p 121
INSTRUMENT	GAL 103 GAL 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2398RG01 14 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001620

04/10/97

LOT RG 2398/01  
hardness - 18 kg

1. Preparation of the dissolution medium  
see page 118

2. Filling the dissolution vessels Bal GAL 111  
1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.1 g	[initials] 04/10/97 [initials] 04/10/97
2	yes	1001.0 g	[initials] 04/10/97 [initials] 04/10/97
3	yes	1001.1 g	[initials] 04/10/97 [initials] 04/10/97
4	yes	1001.0 g	[initials] 04/10/97 [initials] 04/10/97
5	yes	1001.0 g	[initials] 04/10/97 [initials] 04/10/97
6	yes	1001.0 g	[initials] 04/10/97 [initials] 04/10/97

		<u>Conditions</u>	
Dissolutest	T° = 37°C±0.5	[initials] 4/10/97	[initials] 04/10/97
GAL 091	speed 75 rpm	[initials] 4/10/97	[initials] 04/10/97

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

[initials] 07/03/97

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FOURNIER 1001621

4. Readings

Spectro GAL 233

Chronometer GAL 124

LF 178 TER LOT RG 2398/01 18 KG

4/10/1997 3:55 PM

Lambda	No.	Value_E
--------	-----	---------

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001622

4. Readings

Spectro GAL 233

Chronometer GAL 124

LF 178 TER LOT RG 2398/01 18 KG

4/10/1997 3:55 PM

Lambda No. Value\_E  
 Lambda No. Value\_E  
 [See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

**DISSOLUTION**

m:\commun\ging\traidon\distem5  
 date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D LECRIT	TITLE	LF 178 TER RG 2398/01 at 18 KG
DATE	04/10/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 125
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2398RG01 18 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[initials] 07/03/97

Highly confidential subject to protective order

FOURNIER 1001623

290.0 30 1.1910\_1 290.0 59 1.3745\_1  
290.0 60 1.3696\_1

**DISSOLUTION**

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR D.LECRIT TITLE LF 178 TER RG 2398/01 at 18 KG  
DATE 04/10/97 NOTEBOOK NO. LF 178 TER no. 1 p 125  
INSTRUMENT GAL 233 GAL 091 FILE m:\commun\ginq\donnbases\lf178ter\dissolution\lot 2398RG01 18 kg  
WAVELENGTH 290 nm ELUANT 0.025M NaLS  
CUVETTE in mm 2 STIRRING 75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
quantity of active substance						

Control 100 mg/l

DISSOLUTION READINGS

[initials] 07/03/97

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001624

4/10/97

Assay of fenofibrate in  
mixtures prior to compression  
lot 2397/01 RG  
lot 2398/01 RG

Assay done as per procedure 02DAP004-06/05

Chromatographic conditions see p. 99

1. Preparation of the control solutions (to be prepared twice)

\* Stock solutions for controls: balance no. 5 DAP

Introduce about 25.00 mg of accurately weighed fenofibrate arr1241 into a 50 ml flask.

Add 5 ml of THF.

Qsp with ACN.

Place in magnetic stirrer for 30 minutes.

\*\*\*\* 04/10/97 10:26 AM \*\*\*

=====

Sample: FENOFIBRATE  
Control No.: ARR 1241 T1  
Operator: AG

Tare weight: 7.25194 g

Gross weight: 7.27727 g

NET WEIGHT: 0.02533 g

\*\*\*\* 04/10/97 10:29 AM \*\*\*\*

=====

Sample: FENOFIBRATE  
Control No.: ARR 1241 T2  
Operator: AG

Tare weight: 11.07059 g

Gross weight: 11.09549 g

NET WEIGHT: 0.02490 g

which is TM1

which is TM2

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001625

\* Working solutions for controls

Using a graduated pipette, introduce 2 ml of the stock control solution into 20 ml flask  
qsp with mobile phase then homogenize.

yielding:

T1 at 0.5066 µg/10 µl

T2 at ~~0.0~~ 0.498 µg/10 µl

2. Preparation of test solutions (balance no. 3 DAP)

2.1 Stock solutions for tests

Accurately weigh about 350 mg of mixture in 150 ml flask.

\*\*\*\* 04/10/97 08:33 AM \*\*\*

=====

Sample: LF 178TER  
Control No.: 2397RG01 1  
Operator: AG

Tare weight: 14.24833 g

Gross weight: 14.60254 g

NET WEIGHT: 0.35421 g

\*\*\*\* 04/10/97 08:39 AM \*\*\*\*

=====

Sample: LF 178TER  
Control No.: 2398RG01 3  
Operator: AG

Tare weight: 11.96462 g

Gross weight: 12.31560 g

NET WEIGHT: 0.35098 g

\*\*\*\* 04/10/97 08:37 AM \*\*\*

=====

Sample: LF 178TER  
Control No.: 2397RG01 2  
Operator: AG

Tare weight: 15.38274 g

Gross weight: 15.73109 g

NET WEIGHT: 0.34835 g

\*\*\*\* 04/10/97 08:42 AM \*\*\*\*

=====

Sample: LF 178TER  
Control No.: 2398RG01 4  
Operator: AG

Tare weight: 13.15772 g

Gross weight: 13.50633 g

NET WEIGHT: 0.34861 g

Add 15 ml of THF then 7.5 ml of mobile phase.

Place in magnetic stirrer for 45 min.

Qsp with ACN.

Place in magnetic stirrer for 45 min.

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001626



## 2.2. Working solutions for tests

Using a graduated pipette, introduce 1 ml of stock control solution into a 25 ml flask.  
Qsp with mobile phase.  
Homogenize.

## 3. Injections and results

The chromatograms are in appendix 2.

\* Calculating the calibration factor for T1 and the coefficient of variation.

area  [See original for figures.]	$F_{T1} = \frac{\text{feno concn}}{\text{area}}$	[initials] 07/02/97  [equations]  mean of last 3 tests  [equation]
---	--	--

\* calculating the calibration factor for T2

Area  $F_{T2}$

[See original for figures and equation.]

\* verification of controls

$$\frac{F_{T2}}{F_{T1}} \times 100 = \frac{1.468 \times 10^{-6} \times 100}{1.469 \times 10^{-6}} = 99.9\% \text{ controls ok accepted}$$

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001627

\* mean calibration factor =

$$MF = \frac{F_{T1} + F_{T2}}{2} = \frac{(1.468 + 1.469) \times 10^{-6}}{2} = 1.469 \times 10^{-6}$$

\* dose of fenofibrate per tablet  
see procedure for expanded formula

$$375 \times \frac{MF \times \text{area}_{\text{test}} \times 694.4}{PE_{\text{test}}}$$

[see original for formulas]

lot 2397/01 RG at 156 mg of feno/tablet

[see original for formulas]

lot 2398/01 RG at 155 mg of feno/tablet

[initials] 07/09/9[text cut off]

Highly confidential subject to protective order

FOURNIER 1001628

[date cut off]

LOT RG 2399/01

hardness - 15 kg

1. Preparation of the dissolution medium

\* weighing the water Bal GAL 014

tare = 2.730 kg [initials] 04/11/97 [initials] 04/11/97

gross = 25.630 kg [initials] 04/11/97 [initials] 4/11/97

net = 25.630 - 2.730 = 22.900 kg

\* weighing the NaLS Bal GAL 011

tare = 153.5 g then reset [initials] 04/11/97 [initials] 04/11/97

net = 165.1 g [initials] 04/11/97 [initials] 4/11/97

Amount to be weighed for a medium of 0.025M (MW = 288.4 g)

mass of NaLS = 22.[text obscured] = 165.1 g [initials] 4/11/97

2. Filling the [text obscured] vessels [text obscured] GAL 111

1 liter of [text obscured] NaLS [text obscured] g

vessel	reset	[text obscured by weight slip]	verifiers	
1	yes		[initials] 04/11/97	[initials] 04/11/97
2	yes		[initials] 04/11/97	[initials] 04/11/97
3	yes		[initials] 04/11/97	[initials] 04/11/97
4	yes		[initials] 04/11/97	[initials] 04/11/97
5	yes		[initials] 04/11/97	[initials] 04/11/97
6	yes		[initials] 04/11/97	[initials] 04/11/97

Conditions

4/11/97

Dissolutest T° [text obscured] 4/[text obscured]/97 [initials] 37°C±0.[text cut off]  
GAL 091 speed [text obscured] 4/[text obscured]/97 [initials] 4/11/97

3. Weighing the tablets

Bal GAL 205

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001629

[date cut off]

LOT RG 2399/01

hardness - 15 kg

3. Preparation of the dissolution medium

\* weighing the water Bal GAL 014

tare = 2.730 kg [initials] 04/11/97 [initials] 04/11/97

gross = 25.630 kg [initials] 04/11/97 [initials] 4/11/97

net = 25.630 - 2.730 = 22.900 kg

\* weighing the NaLS Bal GAL 011

tare = 153.5 g then reset [initials] 04/11/97 [initials] 04/11/97

net = 165.1 g [initials] 04/11/97 [initials] 4/11/97

Amount to be weighed for a medium of 0.025M (MW = 288.4 g)

mass of NaLS = 22.900 x 288.4 x 0.025 = 165.1 g [initials] 4/11/97

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
2	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
3	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
4	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
5	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
6	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97

Conditions

4/11/97

Dissolutest T° 37°C±0.5 [initials] 04/11/97 [initials] 37°C±0.[text cut off]

GAL 091 speed 75 rpm [initials] 04/11/97 [initials] 4/11/97

3. Weighing the tablets

Bal GAL 205

[text obscured]

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001630

4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2399/01 15 KG

4/11/1997 12:32 PM

Lambda . No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

[initials and date obscured]

Highly confidential subject to protective order

FOURNIER 1001631

#### 4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2399/01 15 KG

4/11/1997 12:32 PM

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

#### DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

#### OPERATING CONDITIONS AND SETTINGS

OPERATOR	D LECRIT	TITLE	LF 178 TER RG 2399/01 at 15 KG
DATE	04/11/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 131
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2399RG01 15 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

#### SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample					
quantity of active substance					

Control 100 mg/l

#### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001632

290.0 30 1.2059\_1 290.0 60 1.3827\_1

**DISSOLUTION**

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2399/01 at 15 KG
DATE	04/11/97	NOTEBOOK NO.	LF 178 TER no. 1 p 131
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2399RG01 15 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

DISSOLUTION READINGS

[initials] 07/09/97

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001633

4/11/97

Assay of the fenofibrate contained in  
tablets of LF 178 ter  
mixture lot 2399/01 RG

Assay done as per procedure 02DAP004-06/05

Chromatographic conditions see p. 99

1. Preparation of the control solutions (to be prepared twice)

\* Stock solutions for controls: balance no. 5 DAP

Introduce about 25.00 mg of accurately weighed fenofibrate arr-171241 into a 50 ml flask.

Add 5 ml of THF

Qsp with ACN.

Magnetic stirring for 30 minutes.

\*\*\*\* 04/11/97 8:46 AM \*\*\*

=====

Sample: FENOFIBRATE  
Control No.: ARR 1241 T1  
Operator: AG

Tare weight: 11.51415 g

Gross weight: 11.53883 g

NET WEIGHT: 0.02468 g

\*\*\*\* 04/11/97 8:48 AM \*\*\*\*

=====

Sample: FENOFIBRATE  
Control No.: ARR 1241 T2  
Operator: AG

Tare weight: 10.87260 g

Gross weight: 10.89800 g

NET WEIGHT: 0.02540 g

which is TM1 and TM2

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001634



4/11/97

\* Working solutions for controls

using a graduated pipette, introduce 2 ml of stock control solution into 20 ml flask  
Qsp with mobile phase.  
Homogenize.

yielding:

T1 at 0.4936 µg/10 µl

T2 at 0.5080 µg/10 µl

2. Preparation of test solutions (balance no. 5 DAP)

2.1 Stock solutions for tests

Accurately weigh about 350.00 mg of mixture in 150 [text cut off] flask.

\*\*\*\* 04/11/97 08:30 AM \*\*\*

\*\*\*\* 04/11/97 08:33 AM \*\*\*\*

=====

Sample: LF 178TER

Control No.: 2399RG01

1

Operator: AG

Tare weight: 18.88493 g

Gross weight: 19.23615 g

NET WEIGHT: 0.35122 g

=====

Sample: LF 178TER

Control No.: 2399RG01

2

Operator: AG

Tare weight: 17.79002 g

Gross weight: 18.14293 g

NET WEIGHT: 0.35291 g

Add 15 ml of THF + 7.5 ml of mobile phase.

Place in magnetic stirrer for 45 min.

Qsp with ACN.

Place in magnetic stirrer for 45 min.

2.2. Working solutions for tests

Using a graduated pipette, introduce 1 ml of stock control solution into a 25 ml flask.

Qsp with mobile phase.

Homogenize.

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001635

### 3. Injections and results

The chromatograms are in appendix 2.

\* Calculating the coefficient of variation and the calibration fact for T1:

area	$F_{T1} = \frac{\text{feno concen of T1}}{\text{area}}$ <p>[initials] 07/09/97</p> <p>[See original for figures.]</p>	
------	---	--

\* calibration factor for T2

Area  $F_{T2}$

[See original for figures and equation.]

\* verification of controls

$$\frac{F_{T2}}{F_{T1}} \times 100 = \frac{1.480 \times 10^{-6}}{1.477 \times 10^{-6}} = 100.2\% \text{ controls ok accepted}$$

\* mean calibration factor

$$MF = \frac{F_{T1} + F_{T2}}{2} = \frac{1.480 + 1.477}{2} = 1.479 \times 10^{-6}$$

\* dose of fenofibrate per tablet

see procedure for expanded formula

$$375 \times \frac{MF \times \text{area}_{\text{test}} \times 694}{P_{\text{test}}} \text{ [illegible]}$$

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001636

[See original for formulas.]

lot 2399/01 RG = 163 mg/tablet

4/11/97

Dissolution - tablet LF178ter  
lot 2399/01 RG  
hardness - 20kg

1. Preparation of the 0.025M NaLS

See p130

2. Weighing the dissolution medium gal 111

1 L of 0.025M NaLS = 1001.0 g

vessel	reset	weight	signature
1	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
2	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
3	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
4	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
5	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97
6	yes	1001.0 g	[initials] 04/11/97 [initials] 04/11/97

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001637

Conditions  
T° 37°C±0.5 [initials] 04/11/97 [initials] 4/11/97  
speed 75 rpm [initials] 04/11/97 [initials] 4/11/97

3. Weighing the tablets bal GAL 205

[See original for weight slip.]

4. Readings Chronometer GAL 124

LF 178 TER LOT RG 2399/01 20 KG
---------------------------------

4/11/1996 2:41 PM

	Lambda	No.	Value_E
Lambda	No.	Value_E	
			[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2399/01 at 20 KG
DATE	04/11/97	NOTEBOOK NO.	178 TER no. 1 p 136
INSTRUMENT	GAL 103 GAL 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2399RG01 20 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001639

04/14/97

Lot RG 2400/01

14 kg

4. Preparation of the dissolution medium

- weighing the water Bal GAL 014

tare = 2.740 kg [initials] 04/14/97 [initials] 04/14/97

gross = 19.780 kg [initials] 04/14/97 [initials] 04/14/97

net = 19.780 - 2.740 = 17.040 kg

- weighing the NaLS Bal GAL 011

for a medium of 0.025M =>

mass of NaLS = 17.040 x 0.025 x 288.4 = 122.9 g

tare = 153.4 g then reset [initials] 04/14/97 [initials] 04/14/97

net = 122.9 g [initials] 04/14/97 [initials] 04/14/97

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.1 g	[initials] 04/14/97 [initials] 04/14/97
2	yes	1001.1 g	[initials] 04/14/97 [initials] 04/14/97
3	yes	1001.0 g	[initials] 04/14/97 [initials] 04/14/97
4	yes	1001.0 g	[initials] 04/14/97 [initials] 04/14/97
5	yes	1001.0 g	[initials] 04/14/97 [initials] 04/14/97
6	yes	1001.0 g	[initials] 04/14/97 [initials] 04/14/97

Conditions

T° 37°C±0.5

[initials] 04/14/97 [initials] 04/14/97

speed 75 rpm

[initials] 04/14/97 [initials] 04/14/97

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001640

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

5. forgot to print the  
reset [initials] 04/14/97

6. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2400/01 14 KG

4/14/1997 12:29 PM

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001641

# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2400/01 at 14 KG
DATE	04/14/97	NOTEBOOK NO.	LF 178 TER no. 1 p 138
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2400RG01 14 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001642



04/14/97

Lot RG 2400/01  
hardness – 18 kg

4. Preparation of the dissolution medium  
See page 138

2. Filling the dissolution vessels Bal GAL 111  
1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.0 g	[initials] 04/14/97 [initials] 04/14/[text cut off]
2	yes	1001.0 g	[initials] 04/14/97 [initials] 04/14/[text cut off]
3	yes	1001.1 g	[initials] 04/14/97 [initials] 04/14/[text cut off]
4	yes	1001.1 g	[initials] 04/14/97 [initials] 04/14/[text cut off]
5	yes	1001.1 g	[initials] 04/14/97 [initials] 04/14/[text cut off]
6	yes	1001.0 g	[initials] 04/14/97 [initials] 04/14/[text cut off]

Conditions

T° 37°C±0.5  
speed 75 rpm

[initials] 04/14/97 [initials] 04/14/97  
[initials] 04/14/97 [initials] 04/14/97

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001643

4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2400/01 18 KG

4/14/1997 4:04 PM

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

[initials and date obscured]

Highly confidential subject to protective order

FOURNIER 1001644

#### 4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2400/01 18 KG

4/14/1997 4:04 PM

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

#### DISSOLUTION

m:\commun\ging\traitdon\distern5  
date printed: 02/06/97

#### OPERATING CONDITIONS AND SETTINGS

OPERATOR	D LECRIT	TITLE	LF 178 TER RG 2400/01 at 18 KG
DATE	04/14/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 142
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2400RG01 18 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

#### SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

#### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001645

290.0 30 1.1981\_1 290.0 60 1.3871\_1

**DISSOLUTION**

m:\commun\ginq\traitdon\distern5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2400/01 at 18 KG
DATE	04/14/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 142
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbases\lf178ter\dissolution\lot 2400RG01 18 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

DISSOLUTION READINGS

[initials] 07/09/97

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001646

04/15/97

LOT RG 2401/01

hardness - 15 kg

1. Preparation of the dissolution medium

- weighing the water Bal GAL 014

tare = 2.740 kg [initials] 04/15/97 [initials] 4/15/97

gross = 25.490 kg [initials] 04/15/97 [initials] 4/15/97

net = 25.490 - 2.740 = 22.750 kg

- weighing the NaLS Bal GAL 011

for a medium of 0.025M =>

mass of the NaLS = 22.750 x 0.025 x 288.4 = 164.0 g

tare = 153.3 g then reset [initials] 04/15/97 [initials] 04/15/97

net = 164.0 g [initials] 04/15/97 [initials] 04/15/97

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.1 g	[initials] 04/15/97 [initials] 04/15/97
2	yes	1001.1 g	[initials] 04/15/97 [initials] 04/15/97
3	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
4	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
5	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
6	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97

Conditions

T = 37°C±0.5

[initials] 04/15/97 [initials] 04/15/97

speed 75 rpm

[initials] 04/15/97 [initials] 04/15/97

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001647

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2401/01 15 KG

4/15/1997 12:27 PM

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001648

# DISSOLUTION

m:\commun\ginq\traidon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2401/01 at 15 KG
DATE	04/15/97	NOTEBOOK NO.	LF 178 TER no. 1 p 145
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2401RG01 15 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

\* transcription error of no significant impact

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001649

04/15/97

Lipidil Micro gelcaps  
Lot 51

1. Preparation of the dissolution medium

See preparation p 143

2. Filling the dissolution vessels

Balance gal 111      1 liter of 0.025M NaLS => 1001.0 g

vessel	reset	weight	signature
1	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
2	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
3	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
4	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
5	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
6	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97

Conditions

[initials] 04/15/97      /      Temperature:= 37°C±0.5°C

[initials] 04/15/97      \      Speed = 75 rpm

Prolabo Dissolutest GAL 103  $\pm$ 290

UV 930 GAL 108 2mm cuvette

5 ml sample collected of the medium and  
replaced with 5 ml of new medium

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001650



3. Weighing the gelcaps [text cut off]

[See original for weight slip.]

4. Readings

LIPIDIL MICRO CANADA GELCAPS LOT 51 0.025M NALS 75 RPM
--

04/15/1996 1:1[text cut off] PM

Lambda No. Value\_E

[See original for figures.]

Comment: Forgot  
samples at  
T= 60 min

Highly confidential subject to protective order

FOURNIER 1001651

[See original for partially obscured weight slip.]

4. Readings

LIPIDIL MICRO CANADA GELCAPS LOT 51 0.025M NALS 75 RPM
--

04/15/1996 1:10 PM

Lambda No. Value\_E

[See original for figures.]

Comment: Forgot  
samples at  
T=60 min

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001652

# DISSOLUTION

m:\commun\ging\traitdon\distern5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	C.COGET	TITLE	lip200 M Canada gelcaps lot 51
DATE	04/15/97	NOTEBOOK NO.	Lf 178ter dissolution no.1 p146
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 51 Canada
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001653

Lot RG 2401/01  
hardness - 20 kg

1. Preparation of the dissolution medium

See page 143

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.1 g	[initials] 04/15/97 [initials] 04/15/97
2	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
3	yes	1001.1 g	[initials] 04/15/97 [initials] 04/15/97
4	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
5	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97
6	yes	1001.0 g	[initials] 04/15/97 [initials] 04/15/97

Conditions

T° 37°C±0.5

[initials] 04/15/97 [initials] 04/15/97

speed 75 rpm

[initials] 04/15/97 [initials] 04/15/97

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001654

4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2401/01 20 KG
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Lambda	No.	Value_E
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[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001655

#### 4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2401/01 20 KG

Lambda No. Value\_E

[See original for figures.]

- 1 AZ [expansion unknown] Air/Air
- 2 NaLS/NaLS
- 3 AZ [expansion unknown] NaLS/NaLS

#### DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

#### OPERATING CONDITIONS AND SETTINGS

OPERATOR	D LECRIT	TITLE	LF 178 TER RG 2401/01 at 20 KG
DATE	04/15/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 150
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2401RG01 20 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

#### SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

#### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[text obscured] 97

Highly confidential subject to protective order

FOURNIER 1001656

290.0 30 1.1368\_1 290.0 60 1.3907\_1

**DISSOLUTION**

m:\commun\ging\traidon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2401/01 at 20 KG
DATE	04/15/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 150
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donbase\lf178ter\dissolution\lot 2401RG01 20 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001657

04/16/97

Lot RG 2402/01  
hardness - 15 kg

1. Preparation of the dissolution medium

- weighing the water Bal GAL 014

tare = 2.730 kg [initials] 04/16/97 [initials] 04/16/97  
gross = 30.360 kg [initials] 04/16/97 [initials] 04/16/97  
net = 30.360 - 2.730 = 27.630 kg

- weighing the NaLS Bal GAL 011

for a medium of 0.025M -> mass of the NaLS to be weighed =  
 $27.630 \times 0.025 \times 288.4 = 199.2 \text{ g}$

SIMPLE WEIGHING

Date weighed: 04/16/1997

08:13:01 AM

\* ARR1768 \*

PRODUCT => NALAUSF

NET WEIGHT => 0.199 KG

TARE => 0.153 KG

GROSS WEIGHT => 0.353 KG

[initials]

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
2	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
3	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
4	yes	1001.1 g	[initials] 04/16/97 [initials] 04/16/97
5	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
6	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97

Conditions

[initials] 07/09/97

[initials] 04/16/97

[initials] 04/16/97

T = 37°C±0.5 [initials] [text cut off]

speed 75 rpm

[initials] [text cut off]

Highly confidential subject to protective order

FOURNIER 1001658



3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2402/01 15 KG
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Lambda No. Value\_E

[See original for figures.]

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001659

# DISSOLUTION

m:\commun\ginq\traidon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2402/01 at 15 KG
DATE	04/16/97	NOTEBOOK NO.	LF 178 TER no. 1 p 153
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2402RG01 15 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001660

04/16/97

Lipidil Micro Gelcaps  
Lot 51  
(second dissolution)

1. Preparation of the dissolution medium

See p 149 151 [initials] 04/16/97

2. Filling the dissolution vessels

balance GAL 111

1 liter of 0.025M NaLS => 1001.0 g

vessel	reset	weight	verifier
1	yes	1001.0 g	[initials] 04/16/97 [initials] 4/16/97
2	yes	1001.0 g	[initials] 04/16/97 [initials] 4/16/97
3	yes	1001.1 g	[initials] 04/16/97 [initials] 4/16/97
4	yes	1001.0 g	[initials] 04/16/97 [initials] 4/16/97
5	yes	1001.1 g	[initials] 04/16/97 [initials] 4/16/97
6	yes	1001.0 g	[initials] 04/16/97 [initials] 4/16/97

Conditions

UV 930 GAL 108  $\lambda=290$  nm  
cuvette 2 mm

Prolabo Dissolutest GAL 103

T° 37°C±0.5°C

75 rpm

[initials] 04/16/97 [initials] 04/16/97

[initials] 07/09/97

Highly confidential subject to protective order

FOURNIER 1001661

## 3. Weighing the gelcaps

balance GAL 205

**DISSOLUTION**m:\commun\ging\traitdon\distern5  
date printed: 02/06/97OPERATING CONDITIONS AND SETTINGS

OPERATOR	C.COGET	TITLE	lip200 M Canada gelcaps lot 51
DATE	04/16/97	NOTEBOOK NO.	Lf 178ter dissolution no.1 p154
INSTRUMENT	gal 103 108	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 51 Canada test 2
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/09/[text cut off]

Highly confidential subject to protective order

FOURNIER 1001662

3. Weighing the gelcaps balance GAL 205

[See original for weight slip.]

4. Readings and results

LIPIDIL MICRO CANADA GELCAPS LOT 51 0.025M NALS 75 RPM

04/16/1996 10:28 AM

Lambda No. Value\_E

[See original for figures.]

Highly confidential subject to protective order

FOURNIER 1001664

04/16/97  
Lot RG 2402/01  
hardness - 20 kg

1. Preparation of the dissolution medium

See page 151

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifiers
1	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
2	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
3	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
4	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
5	yes	1001.1 g	[initials] 04/16/97 [initials] 04/16/97
6	yes	1001.1 g	[initials] 04/16/97 [initials] 04/16/97

Conditions

T° = 37°C±0.5

[initials] 04/16/97 [initials] 04/16/97

speed = 75 rpm

[initials] 04/16/97 [initials] 04/16/97

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001665

4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2402/01 20 KG
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Lambda	No.	Value_E
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[See original for figures.]

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001666



#### 4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2402/01 20 KG

Lambda No. Value\_E

[See original for figures.]

#### DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

#### OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2402/01 at 20 KG
DATE	04/16/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 157
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2402RG01 20 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

#### SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

#### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Highly confidential subject to protective order

FOURNIER 1001667

290.0 30 1.1566\_1 290.0 60 1.4079\_1

**DISSOLUTION**

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2402/01 at 20 KG
DATE	04/16/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 157
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2402RG01 20 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001668

4/[cut off, possibly 1]6/97

Lipidil Micro Gelcaps  
Lot 52

1. Preparation of the dissolution medium

See page 151

2. Filling the dissolution vessels

1 liter of 0.025M NaLS => 1001.0 g to be weighed

vessel	reset	weight	verifiers
1	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
2	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
3	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
4	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
5	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97
6	yes	1001.0 g	[initials] 04/16/97 [initials] 04/16/97

Conditions:

UV 930 GAL 108  $\lambda=290$  nm

cuvette 2 mm

Prolabo Dissolutest GAL 103

37°C±0.5°C [initials] 04/16/97 [initials] 04/16/97

75 rpm [initials] 04/16/97 [initials] 04/16/97

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001669

3. Weighing the gelcaps balance GAL 205

[See original for weight slip.]

4. Readings

LIPIDIL MICRO CANADA GELCAPS LOT 52 0.025M NALS 75 RPM
--

04/16/1996 3:13 PM

Lambda No. Value\_E

[See original for figures.]

Highly confidential subject to protective order

FOURNIER 1001670

[This page is inverted.]

[See original for figures.]

04/16/1996 3:13 PM

Lambda No. Value\_E

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001671

# DISSOLUTION

m:\commun\ginq\traidon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	C.COGET	TITLE	lip200 M Canada gelcaps lot 52
DATE	04/16/97	NOTEBOOK NO.	Lf 178ter dissolution no.1 p158
INSTRUMENT	gal 103 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 52 Canada
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001672

04/17/97

Lot RG 2403/01  
hardness - 15 kg

1. Preparation of the dissolution medium

- weighing the water Bal GAL 014

tare = 2.725 kg [initials] 04/17/97 [initials] 04/17/97  
gross = 23.890 kg [initials] 04/17/97 [initials] 04/17/97  
net = 23.890 - 2.725 = 21.165 kg

- weighing the NaLS Bal GAL 011

for a medium of 0.025M, mass of the NaLS to be weighed  
= 21.165 x 0.025 x 288.4 = 152.6 g

SIMPLE WEIGHING

Date weighed: 04/17/1997

08:11:18 AM

\* ARR1768 \*

PRODUCT => NALAUSF

NET WEIGHT => 0.153 KG

TARE => 0.154 KG

GROSS WEIGHT => 0.306 KG [initials]

2. Filling the dissolution vessels Bal GAL 111

1 liter of 0.025M NaLS -> 1001.0 g

vessel	reset	mass of 0.025M NaLS	verifier
1	yes	1001.1 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
2	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
3	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
4	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
5	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
6	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]

Conditions

T° 37°C±0.5 [initials] 04/17/97 [initials] 04/17/97  
speed 75 rpm [initials] 04/17/97 [text cut off]

Highly confidential subject to protective order

FOURNIER 1001673

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

4. Readings

Chronometer GAL 124

LF 178 TER LOT RG 2403/01 15 KG

Lambda No. Value\_E

[See original for figures.]

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001674



# DISSOLUTION

m:\commun\ginq\traitdon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2403/01 at 15 KG
DATE	04/17/97	NOTEBOOK NO.	LF 178 TER no. 1 p 163
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2403RG01 15 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001675

04/17/97

Lipidil Micro Gelcaps  
Lot 53

1. Preparation of the dissolution medium

See page 161

2. Filling the dissolution vessels

1 liter of 0.025M NaLS => 1001.0 g to be weighed

vessel	reset	weight	verifier
1	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/97
2	yes	1001.1 g	[initials] 04/17/97 [initials] 04/17/97
3	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/97
4	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/97
5	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/97
6	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/97

Conditions

UV 930 GAL 108  $\lambda=290$  nm  
cuvette 2 mm

Prolabo Dissolutest GAL 103

37°C $\pm$ 0.5°C [initials] 04/17/97 [initials] 04/17/97  
75 rpm [initials] 04/17/97 [initials] 04/17/97

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001676

3. Weighing the gelcaps

balance AG204 GAL 205

[See original for weight slip.]

4. Readings

LIPIDIL MICRO CANADA GELCAPS LOT 53 0.025M NALS 75 RPM

04/17/1996 11:[text cut off] AM

Lambda No. Value\_E

[See original for figures.]

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001677

[page inverted - See original for figures.]

04/17/1996 11:30 AM

Lambda	No.	Value_E
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Highly confidential subject to protective order

FOURNIER 1001678

# DISSOLUTION

m:\commun\ginq\traitdon\distern5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	C.COGET	TITLE	lip200 M Canada gelcaps lot 53
DATE	04/17/97	NOTEBOOK NO.	Lf 178ter dissolution no.1 p164
INSTRUMENT	gal 103 108	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 53 Canada
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test sample						
quantity of active substance						

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time [initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001679

04/17/97

Lot RG 2403/01  
Hardness – 19 kg

4. Preparation of the dissolution medium  
See page 161

2. Filling the dissolution vessels Bal GAL 111  
1 liter of 0.025M NaLS => 1001.0 g

vessel	reset	mass of the 0.025M NaLS	verifier
1	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
2	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
3	yes	1001.1 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
4	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
5	yes	1001.1 g	[initials] 04/17/97 [initials] 04/17/[text cut off]
6	yes	1001.0 g	[initials] 04/17/97 [initials] 04/17/[text cut off]

Conditions  
T° 37°C±0.5 [initials] 04/17/97 [initials] 04/17/97  
speed 75 rpm [initials] 04/17/97 [initials] 04/17/97

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001680

4. Readings

Bal ~~GAL 205~~ Chronometer GAL 124  
[initials]

LF 178 TER LOT RG 2403/01 19 KG

7/17/1997 4:00 PM

Lambda No. Value\_E

[See original for figures.]

[initials and date obscured]

Highly confidential subject to protective order

FOURNIER 1001681

4. ReadingsBal ~~GAL~~ 205 Chronometer GAL 124  
[initials]

LF 178 TER LOT RG 2403/01 19 KG

4/17/1997 4:00 PM

Lambda No. Value\_E

[See original for figures.]

## DISSOLUTION

m:\commun\ging\traitdon\distem5  
date printed: 02/06/97OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2403/01 at 19 KG
DATE	04/17/97	NOTEBOOK NO.	LF 178 TER no. 1 p. 168
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 2403RG01 19 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001682



[cut off]

290.0 30 1.1864\_1 290.0 60 1.3746\_1

**DISSOLUTION**

m:\commun\ginq\traitdon\distern5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	LF 178 TER RG 2403/01 at 19 KG
DATE	04/17/97	NOTEBOOK NO.	LF 178 TER no. 1 p 168
INSTRUMENT	GAL 233 GAL 091	FILE	m:\commun\ginq\donnbase\lf178ter\dissolution\lot 2403RG01 19 kg
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	694.4	
theoretical dosage	160	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

DISSOLUTION READINGS

[initials] 07/10/97

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001683

4/24/97

Dissolution of LF 178 ter CANADA gelcaps  
lot 34

1. Preparation of the 0.025M NaLS dissolution medium

molecular mass of the ~~0.025~~ NaLS 288.4 g

05/14/97

Dissolution of Lipidil 200 Micro gelcaps  
Lot 34 Canada

1. Preparation of the dissolution medium

\* water

or measure 15 liters using the 5 liter volumetric flask

quantity measured: 15 liters [initials] 05/14/97 [initials] 05/14/97

\* weighing the NaLS Bal 111

For a 0.025M medium, mass of NaLS to be

weighed =  $15 \times 0.025 \times 288.4 = 108.2$  g

reset = calibration [initials] 05/14/97 [initials] 05/14/97

amount weighed = 108.2 g [initials] 05/14/97 [initials] 05/14/97

NaLS lot: 961620 1682

Control: 0020787 exp 12/10/97

2. Filling the dissolution vessels

1 liter of 0.025M NaLS per vessel, measured using a  
volumetric flask.

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001685

vessel	quantity measured	verifiers
1	1 liter	[initials] 05/14/97 [initials] 05/14/97
2	1 liter	[initials] 05/14/97 [initials] 05/14/97
3	1 liter	[initials] 05/14/97 [initials] 05/14/97
4	1 liter	[initials] 05/14/97 [initials] 05/14/97
5	1 liter	[initials] 05/14/97 [initials] 05/14/97
6	1 liter	[initials] 05/14/97 [initials] 05/14/97

Dissolution conditions

T° 37°C±0.5 [initials] 5/15/97 [initials] 05/15/97

speed ~~120 rpm~~ [initials] 05/14/97

speed 75 rpm [initials] 5/15/97 [initials] 05/15/97

Dissolutest DAP: Sotax 6

3. Weighing the tablets Bal GAL 205

[See original for weight slip.]

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001686

4. Readings

Uvikon 942 Chronometer GAL 122

LIPIDIL MICRO 200 LOT 34 CANADA

5/15/1997 10:57 AM

	Lambda	No.	Value_E
Lambda	No.	Value_E	

[See original for figures.]

[date and initials obscured]

Highly confidential subject to protective order

FOURNIER 1001687

#### 4. Readings

Uvikon 942 Chronometer GAL 122

LIPIDIL MICRO 200 LOT 34 CANADA

5/15/1997 10:57 AM

Lambda No. Value\_E  
Lambda No. Value\_E

[See original for figures.]

#### DISSOLUTION

m:\commun\ging\traitdon\distern5  
date printed: 02/06/97

#### OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	lipidil micro 200 Canada gelcaps lot 34
DATE	05/15/97	NOTEBOOK NO.	Lf 178ter dissolution no.1 p170
INSTRUMENT	Sotax 6 - Uvikon 942	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 34 Canada
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

[See original for figures.]

#### SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

mass of test sample  
quantity of active substance

[initials] 07/11/97

Control 100 mg/l

#### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Highly confidential subject to protective order

FOURNIER 1001688

290.0 30 0.9662\_1 [readings cut off]

**DISSOLUTION**

m:\commun\ging\traidon\distem5  
date printed: 02/06/97

OPERATING CONDITIONS AND SETTINGS

OPERATOR	D.LECRIT	TITLE	lipidil micro 200 Canada gelcaps lot 34
DATE	05/15/97	NOTEBOOK NO.	Lf 178ter dissolution no.1 p170
INSTRUMENT	Sotax 6 - Uvikon 942	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 34 Canada
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2 mm	STIRRING	75 rpm

[See original for figures.]

SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

mass of test sample	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
quantity of active substance						

Control 100 mg/l

DISSOLUTION READINGS

[initials] 07/11/97

volume sampled in ml	TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Not redone because not a significant difference [initials] 07/11/97

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

Highly confidential subject to protective order

FOURNIER 1001689

May 15, 1997

Dissolution of Lipidil Micro 200 gelcaps  
Lot 35 Canada

1. Preparation of the dissolution medium 0.025 NaLS

see p. 170

3. Filling the dissolution vessels

1 liter of 0.025M NaLS measured using a volumetric flask.

Vessel	quantity measured	verifiers
1	1 liter	[initials] 05/15/97 [initials] 5/15/97
2	1 liter	[initials] 05/15/97 [initials] 5/15/97
3	1 liter	[initials] 05/15/97 [initials] 5/15/97
4	1 liter	[initials] 05/15/97 [initials] 5/15/97
5	1 liter	[initials] 05/15/97 [initials] 5/15/97
6	1 liter	[initials] 05/15/97 [initials] 5/15/97

3. Dissolution conditions

Dissolutest DAP: Sotax 6

T° = 37°C±0.5°C [initials] 05/15/97 [initials] 5/15/97

speed = 75 rpm [initials] 05/15/97 [initials] 5/15/97

3. Weighing the tablets gal 205

[See original for weight slip.]

[initials] [date illegible]

Highly confidential subject to protective order

FOURNIER 1001690



LIPIDIL MICRO 200 LOT 34 CANADA

5/15/1997 2:20 PM

Lambda No. Value\_E

[See original for figures.]

[initials] 07/10/97

Highly confidential subject to protective order

FOURNIER 1001691

# DISSOLUTION

m:\commun\ging\traidon\distem5  
date printed: 02/06/97

## OPERATING CONDITIONS AND SETTINGS

OPERATOR	A GRANDJEAN	TITLE	lipidil micro 200 Canada gelcaps lot 35
DATE	05/15/97	NOTEBOOK NO.	Lf 178ter dissolution no.1 p172 9
INSTRUMENT	Sotax 6 - Uvikon 942	FILE	m:\commun\ging\donnbase\lf178ter\dissolution\lot 35 Canada
WAVELENGTH	290 nm	ELUANT	0.025M NaLS
CUVETTE in mm	2mm	STIRRING	75 rpm

[See original for figures.]

## SAMPLE PREPARATION

theoretical mass	200	
theoretical dosage	200	in mg

mass of test sample  
quantity of active substance

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6

Control 100 mg/l

## DISSOLUTION READINGS

volume sampled in ml		CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
TIME POINT	volume in ml						

## RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

## RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV

% dissolved

quantity dissolved

[See original for graphs.]

time

time

[initials]07/10/97

Highly confidential subject to protective order

FOURNIER 1001692

[initials] 07/10/97 - Closed

Highly confidential subject to protective order

FOURNIER 1001693

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Highly confidential subject to protective order

FOURNIER 1001695

111	04/08/97 Assay of fenofibrate in MF 2394/01 RG and 2396/01 RG
116	04/09/97 Dissolution - 2397/01 RG 14 kg
118	04/10/97 Dissolution - 2397/01 RG 18 kg
121	04/10/97 Dissolution 2398/01 RG - 14 kg
124	04/10/97 Dissolution 2398/01 RG - 18 kg
126	04/10/97 Assay of fenofibrate in MF - 2397/01 RG - 2398/01 RG
130	04/11/97 Dissolution - 2399/01 RG - 15 kg
132	04/11/97 Assay of fenofibrate in MF - 2399/01 RG
135	04/11/97 Dissolution - 2399/01 RG - 20 kg
138	04/14/97 Dissolution - 2400/01 RG - 14 kg
141	04/14/97 Dissolution - 2400/01 RG - 18 kg
143	04/15/97 Dissolution - 2401/01 RG - 15 kg
146	04/15/97 Dissolution - Lipidil Micro - lot 51
149	04/15/97 Dissolution - 2401/01 RG - 20 kg
151	04/16/97 Dissolution - 2402/01 RG - 15 kg
154	04/16/97 Dissolution - Lipidil Micro - lot 51
156	04/16/97 Dissolution - 2402/01 RG - 20 kg
158	04/16/97 Dissolution - Lipidil Micro - lot 52
161	04/17/97 Dissolution - 2403/01 RG - 15 kg
164	04/17/97 Dissolution - Lipidil Micro - lot 53
167	04/17/97 Dissolution - 2403/01 RG - 19 kg
170	05/14/97 Dissolution - Lipidil Micro - lot 34
173	05/15/97 Dissolution - Lipidil Micro - lot 35

[initials] 07/10/97 Closed

Highly confidential subject to protective order

FOURNIER 1001696

page	Date	Description
003	02/18/97	Dissolution tests - Tween 80
5 to 21	02/19/97 to 02/24/97	Preparation of fenofibrate solutions Obtaining absorption spectra for set of standards
23	02/26/97	Preparation of a fenofibrate solution in ethanol - absorption spectrum
27	02/26/97	Dissolution in 2% Tween 80 - 75 rpm - lot 340
31	02/27/97	Dissolution in 2% Tween 80 - 75 rpm Lip 200 gelcaps ARR 1710
36	02/26/97	Dissolution - tabl lot 340 - 0.02M NaLS - 75 rpm
040	02/26/97	Dissolution - gelcaps lip 200 - arr1710 - 0.02M NaLS - 75 rpm
043	02/28/97	Dissolution - tabl lot 340 - 0.025M NaLS 0-75 rpm
047	03/04/97	Dissolution - gelcaps lip 200 - arr1710 - 0.025M NaLS - 75 rpm
052	03/06/97	Saturation concentration of co-micronizate in 0.025 NaLS
055	03/10/97	Dissolution lot 351 - 0.025M NaLS - 75 rpm
058	03/11/97	Dissolution lot 354 - 0.025M NaLS - 75 rpm
061	03/11/97	Dissolution lot 358 - 0.025M NaLS - 75 rpm
064	03/13/97	Dissolution lot 361 - 0.025 NaLS - 75 rpm
068	03/14/97	Dissolution lot 334 - 0.025 NaLS - 75 rpm
072	03/20/97	Dissolution Lipidil 200 - lots 48,49,50 0.025M NaLS - 75 rpm
081	03/25/97	Dissolution Lipanthyl M - lot 2177 " "
085	04/03/97	Dissolutions LF178TER lot 2394/01RG - 14 and 18 kg " "
092	04/07/97	Dissolution 2393/01RG Opadry OYB
095	04/07/97	Dissolution - 2394/01RG - 14 kg
097	04/07/97	Dissolution - 2394/01RG - 18 kg
099	04/07/97	Assay of fenofibrate lot 2394/01RG - 14 kg and 18 kg
104	04/08/97	Dissolution lot 2396/01RG - 18 kg
106	04/08/97	Dissolution 2393/01RG coated with Opadry - 15 days at 75% RH
109	04/08/97	Dissolution - lot 2396/01 RG 14 kg

Highly confidential subject to protective order

FOURNIER 1001697

I, the undersigned, Patrick NOURISSAT, esquire, Notary and Associate with the Partnership "Patrick Nourissat, Didier Nourissat, and Hugues Misserey," and holding the position of Notary with an office based in DIJON, 25, rue Buffon,

Hereby certify and attest that I have on this day closed the present book containing [illegible, possibly 180] pages, to have numbered and initialed it on the last page, intended for use in the registration of drug formulations for the Laboratoires FOURNIER, Biopharmaceutical Science center, 42 rue de Longvic, in Chenôve.

EXECUTED FOR ALL LEGAL INTENTS AND PURPOSES  
IN DIJON  
IN THE YEAR TWO THOUSAND [illegible]  
ON June 11

[seal:]  
Mr. Patrick Nourissat  
Notary<sup>1</sup> and Associate  
Court of Appeals  
DIJON



Exh A



**GEOTEXT**  
Translations, Inc.

STATE OF NEW YORK        )  
                                  )  
                                  )  
COUNTY OF NEW YORK    )

ss

**CERTIFICATION**

This is to certify that the attached translation is, to the best of my knowledge and belief, a true and accurate translation from French into English of "LF 178TER Lab Notebook, Dissolution No. 2."

Randon Burns, Vice President  
Geotext Translations, Inc.

Sworn to and subscribed before me

this 7<sup>th</sup> day of October, 20 04.

**EZECHIEL A. COPIC**  
**Notary Public, State of New York**  
**No. 01C06104132**  
**Qualified in Queens County**  
**Commission Expires January 12, 2008**

LF 178 TER  
Dissolution No. 2

FOURNIER 1001699

Highly Confidential Subject to Protective Order

LF 178 Ter Lab Notebook  
Dissolution No. 2

This notebook comprises 180 pages. There is a pagination anomaly with page 152, which appears twice.

Notebook approved by MT on June 26, 2000.

MT June 26, 2000.

Began on May 15, 1997 and concluded on July 30, 1997.

FOURNIER 1001700

Highly Confidential Subject to Protective Order

Dissolution of 200 Micro Lipidil Capsules  
Lot 37 Canada

1. Preparation of the dissolution medium

- Water

28 liters of water is measured using graduated 5- and 1- liter volumetric flasks.

Quantity measured: 28 liters M May 15, 1997

CC May 15, 1997

- Weight of LSNa Gal [illegible]

For a medium to 0.025 n, mass of LSNa to be weighed=  $28 \times 0.025 \times 288.4 = 201.88\text{g}$

- Tare of beaker M May 15, 1997 [initials] May 15, 1997

- Amount weighed: 201.9 g M May 15, 1997 [initials] May 15, 1997

LSNa Lot 9616201682 Key No.: 0020787

2. Filling of dissolution bowls

1 liter of 0.025 M LSNa per bowl measured using a volumetric flask

[initials] June 24, 1997

FOURNIER 1001701

Bowl	Amount measured	Verified by
[See original for numbers.]	1 liter [repeats throughout column]	CC May 15, 1997 M May 15, 1997 [repeats throughout column]

Dissolution Condition

Temp.= 37°C ± 0.5      [initials] May 16, 1997 M May 16, 1997  
Speed= 75 TPN      [initials] May 16, 1997 M May 16, 1997

DAP Dissolutest: Sotax 5

3. Weight of capsules    Bal Gal 205  
Code: Lot 37 Lipidil Micro 800 Canada  
[See original for numbers.]

4. Reading

Uvikon 810 DAP      Chronometer Gal 122

[illegible initials and date]

FOURNIER 1001702

## DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

### CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Lipidil Micro 200 Capsules Lot 37 Canada
DATE	05/16/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 2
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

### SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for numbers.]					

Control 100  
mg/l

### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

### RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

### RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001703

004

[See original for numbers.]

FOURNIER 1001704

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Lipidil Micro 200 Capsules Lot 37 Canada
DATE	05/16/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 5-002 RC
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							
Calculation sheet reworked with the correct figure M July 8, 1997							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001705



May 16, 2004

Dissolution 200 M Lipidil Capsules Lot 38 Canada
---

1. Preparation of 0.025 M LSNa  
See page 002.

2. Filling of dissolution bowls

1 L of 0.025 M LSNa measured using a 1-liter graduated cylinder

Bowls	Amount measured	Signature
[See original for numbers.]	1 liter [repeats throughout column]	M May 15, 1997 Ab May 15, 1997 [repeats throughout column]

3. Dissolution conditions  
Dissolutest Sotax AT7 DAP No. 6

Temp= 37°C  $\pm$ 0.5      M May 16, 1997      Ab May 16, 1997  
Speed=75 RPM    M May 16, 1997      Ab May 16, 1997

4. Weight of capsules    Gal 205

[initials] June 24, 1997

FOURNIER 1001706

DISSOLUTION

007

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Lipidil Micro 200 Capsules Lot 38 Canada
DATE	05/16/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 6
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001707

[See original for numbers.]

5. Reading  
on 942 DAP Kontron photospectrometer  
2 mm optical view tanks

200M LIPIDIL CAPSULES CANADA LOT 38

Lambda	No.	Value_E
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[See original for table.]

FOURNIER 1001708

008

May 16, 2004

Dissolution 200 M Lipidil Capsules  
Lot 39 Canada

1. Preparation of 0.025 M LSNa  
0.025M LSNa See page 002.

2. Volume measure of bowls

1 L of 0.025 M LSNa measured using a 1-liter graduated cylinder

Bowl	Amount measured	Signature
[See original for numbers.]	1 liter [repeats throughout column]	M May 16, 1997 Ab May 16, 1997 [repeats throughout column]

3. Weight of capsules Gal 205

[See original for numbers.]

[illegible initials and date]

FOURNIER 1001709

4. Operating conditions

Dissolutest Sotax AT7 DAP No. 6

Temp= 37°C ±0.5	Ab May 16, 1997	M May 16, 1997
Speed=75 RPM	Ab May 16, 1997	M May 16, 1997

5. Reading

on DAP Kontron 942 photospectrometer

200M LIPIDIL CAPSULES CANADA LOT 39

Lambda	No.	Value_E
--------	-----	---------

[See original for table.]

[illegible initials and date]

FOURNIER 1001710

010

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Lipidil Micro 200 Capsules Lot 39 Canada
DATE	06/16/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 8
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001711

May 16, 1997

011

Dissolution of 200 Micro Lipidil Capsules  
Lot 40 Canada

1. Preparation of the dissolution medium  
See page 2.

2. Filling of dissolution bowls

1 liter of 0.025 M LSNa per bowl measured using a 1-liter volumetric flask

Bowl	Amount measured	Verified by
[See original for numbers.]	1 liter [repeats throughout column]	M May 16, 1997 VA May 16, 1997 [repeats throughout column]

Dissolution Conditions

Temp.= 37°C ± 0.5      M May 16, 1997 VA May 16, 1997

Speed 420 TP      M May 16, 1997

Speed= 75 TPN      M May 16, 1997 VA May 16, 1997

DAP Dissolutest: Sotax 5

[illegible initials and date]

FOURNIER 1001712

3. Weight of capsules Bal Gal 205  
Code Lot: 40 Lipidil Micro Canada

[See original for table.]

6. Reading

Uvikon 810 DAP                      Chronometer Gal 122

[illegible initials and date]

FOURNIER 1001713



[The text on FOURNIER 1001714 is a duplicate of that on FOURNIER 1001713.]

FOURNIER 1001714

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DISSOLUTION

013

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Lipidil Micro 200 Capsules Lot 37 Canada
DATE	05/16/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 13 OM RC
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001715

Dissolution of LF 178 Ter Tablet Canada Lot CO 194
---

1. Preparation of the dissolution medium, 0.025 M LSNa

A small amount of water is weighed on PM4600 Gal065 balance in a 3 l beaker.

It is poured in to a 10 l beaker.

The corresponding LSNa is weighed. It is mixed into the water in the 10 l beaker.

Water is weighed in a 3 l beaker, and the corresponding LSNa is weighed.

Mix.

Pour the LSNa solutions from the 2 beakers into a 20-liter demijohn. Mix.

\* 10-liter beaker - water

[See original for numbers.]

tare

net

gross

forgot to print gross Ab

total  $1908.9 + 2050.8 + 2137.1 + 3059.3 = 9156.1$  g or 9.156 kg

RC June 6, 1997

FOURNIER 1001716

amount of LSNa [illegible] 1547 to be weighed in 10 l beaker

$9.156 \times 0.025 \times 288.4 = 66.01 \text{ g}$  [illegible] LSNa=288.4g

[See original for numbers.]

tare  
net  
gross

\* 3 l beaker  
water

[See original for numbers.]

i.e.  $3.145 \times 288.4 \times 0.025 = 22.68 \text{ g}$  of LSNa for weighing

tare  
net  
gross

2. Weight of dissolution medium Gal 065  
1 l of 0.025M LSNa=1001.0 g

[See original for numbers.]

tare  
net  
gross

forgot to print gross

[illegible initials and date]

FOURNIER 1001717

016

3. Dissolution conditions  
dissolutest Sotax AT 7 Gal 091

Temp.=37°C ±0.5°C	Ab June 3, 1997	SC June 3, 1997
Speed: 75 RPM	Ab June 3, 1997	SC June 3, 1997

4. Weight of tablets Gal 205

[See original for numbers.]

5. Reading  
on photospectrometer KONTRON 922 Gal 233  
2 mm optical view tanks

Wavelength (nm)  
Wait time (s)  
Integration time (s)  
Number of samples

Calc mode	No
Lamp adjustment (nm)	
Deuterium lamp	Yes
Tungsten lamp	Yes
Fissure (s) (nm)	
Auto print	No
Auto save	No
Auto transfer	No

KONTRON INSTRUMENTS UVIKON 922

Operator

[illegible initials and date]

FOURNIER 1001718

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Tablets Canada Lot CO194
DATE	06/03/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 14
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

RC 6/6/97

FOURNIER 1001719

Fixed Wavelength

Lambda	No.	Value _E
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[See original for table.]

FOURNIER 1001720

June 3, 1997

Dissolution LF178 Ter Tablet Canada Lot Co 197
---

1. Preparation of dissolution medium LSNa 0.025M

See p. 14.

2. Weight of dissolution medium Gal 065

1 liter of 0.025M LSNa= 1001.0g

[See original for numbers.]

tare  
net  
gross

3. Dissolution conditions  
dissolutest Sotax AT 7 Gal 091

Temp.=37°C ±0.5°C  
Speed: 75 RPM

Ab June 3, 1997  
Ab June 3, 1997

SC June 3, 1997  
SC June 3, 1997

4. Weight of tablets Gal 205

RC June 6, 1997

FOURNIER 1001721



[See original for numbers.]

5.     Reading  
          on photospectrometer KONTRON 922 Gal 233  
          2 mm optical view tanks

LF 178 Ter Tablets Canada Lot CO197

Lambda.     No.     Value\_E

[See original for numbers.]

drop [illegible] and invert [illegible] more  
product to redo the reading Ab 6/3

[illegible initials and date]

FOURNIER 1001722

## DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

### CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Tablets Canada Lot CO197
DATE	06/03/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 18
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

### SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for numbers.]					

Control 100  
mg/l

### DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

### RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

### RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

RC 6/6/97

FOURNIER 1001723

June 5, 1997

Dissolution LF 178 Ter Tablet Canada Lot CO 200
--

1. Preparation of dissolution medium 0.025M LSNa  
Gal 065 Balance

Weight of water

[See original for numbers.]

tare for a total of  $2609.2 + 2271.3 + 1655.6 = 6.536$  kg

net

gross

for  $6.536 \times 288.4 \times 0.025 = 47.1$  g of LSNa for weighing

Weight of LSNa

[See original for numbers.]

tare

net

gross

RC June 6, 1997

FOURNIER 1001724

022

2. Weight of dissolution medium Gal 065

1 l of 0.025M LSNa weighs 1001.0g

[See original for numbers.]

tare  
net  
gross

3. Dissolution conditions  
dissolutest Sotax AT 7 Gal 091

Temp.=37°C ±0.5°C  
Speed: 75 RPM

RC June 5, 1997  
RC June 5, 1997

Ab June 5, 1997  
Ab June 5, 1997

4. Weight of tablets Gal 205

Ab June 6, 1997 RC

FOURNIER 1001725

5. Reading  
on photospectrometer KONTRON 922 Gal 233  
2 mm optical view tanks

#### FIXED WAVELENGTH

##### Parameter List

Wavelength (nm)  
Wait time (s)  
Integration time (s)  
Number of samples

Calc mode	No
Lamp adjustment (nm)	
Deuterium lamp	Yes
Tungsten lamp	Yes
Fissure (s) (nm)	
Auto print	No
Auto save	No
Auto transfer	No

KONTRON INSTRUMENTS UVIKON 922

Operator

FIXED WAVELENGTH 290NM 75RPM LF178 Ter LOT CO200

[See original for table.]

Lambda	No.	Value_E
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Ab June 6, 1997

FOURNIER 1001726

024

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Tablets Canada Lot CO200
DATE	05/06/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 21
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001727

June 9, 1997

Dissolution LF 178 Ter Tablets  
Lot PK #158 at 100 mg of Feno

Medium of 0.025M LSNa at 75 RPM

1. Preparation of the dissolution medium Gal 065 Balance

Weight of the water:

WATER

[See original for numbers.]

Tare Total weight of water=

Net

Gross For a 0.025M medium  
Mass of LSNa for weighing

[illegible initials and date]  
FOURNIER 1001728

026

2. Weight of Dissolution Medium Gal 065 Bal  
1 liter of 0.025 M LSNa 1001.0 g

[See original for numbers.]

Tare  
Net  
Gross

3. Dissolution conditions  
dissolutest Sotax AT 7 Gal 091

Temp.=37°C ±0.5°C M June 9, 1997 [initials] June 9, 1997  
Speed: 75 RPM M June 9, 1997 [initials] June 9, 1997

4. Weight of tablets Gal 205

[See original for numbers.]

[illegible initials and date]

FOURNIER 1001729



5. Reading  
on photospectrometer KONTRON 922 Gal 233  
2 mm optical view tanks

LF 178 Ter LOT 158, 0.025M 75 RPM

Lambda.	No.	Value_E
---------	-----	---------

[See original for table.]

[illegible initials and date]

FOURNIER 1001730

028

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	Tablets PK Lot CO200
DATE	06/09/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 28 25 RC
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.025 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001731

Highly Confidential Subject to Protective Order

June 10, 1996

Dissolution LF 178 Ter Tablets  
Lot PK #158 at 100 mg of Feno

Medium of 0.05M LSNa at 120 RPM

1. Preparation of the dissolution medium Gal 065 Balance

Weight of the water:

WATER

[See original for numbers.]

Tare Total weight of water=

Net

Gross Weight of LSNa:  
For a medium of 0.05 M

FOURNIER 1001732

2. Weight of Dissolution Medium Gal 065 Bal  
1 liter of 0.05 M LSNa 1002.0 g

[See original for numbers.]

Tare  
Net  
Gross

3. Dissolution conditions  
dissolutest Sotax AT 7 Gal 091

Temp.=37°C ±0.5°C M June 10, 1997 Ab June 10, 1997  
Speed: 75 RPM M June 10, 1997 Ab June 10, 1997

4. Weight of tablets Gal 205 Bal

[See original for numbers.]

[illegible initials and date]

FOURNIER 1001733

5. Reading  
photospectrometer Gal 233 Chronometer Gal 124  
2 mm tanks - 290nm

LF 178 Ter LOT 158, 0.05M 120 RPM LSNA

Lambda. No. Value\_E

[See original for numbers.]

[illegible initials and date]

FOURNIER 1001734

032

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	LFter Tablets PK Lot 158— 0.05M 120 RPM
DATE	09/09/97 06/10/97 RC	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 32 29 RC
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.05 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							
Not removed so a non-significant difference M July 7, 1997.							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001735

Highly Confidential Subject to Protective Order

June 11, 1997

Dissolution LF 178 Ter Tablets  
Lot PK 158 at 100mg Lot CO194 M June 11, 1997

Medium of 0.01M LSNa at 420 90 RPM M 6/11/97

1. Preparation of the dissolution medium Gal 065 Balance

Weight of the water:

WATER

[See original for numbers.]

Tare Total weight of water:

Net

Gross Weight of LSNa  
For a 0.01M medium

RC June 24, 1997

FOURNIER 1001736

034

2. Weight of Dissolution Medium Gal 065 Bal  
1 liter of 0.025 M LSNa 1004.0 g

[See original for numbers.]

Tare  
Net  
Gross

3. Dissolution conditions  
Dissolutest Gal 091

Temp.=37°C ±0.5°C M June 11, 1997 [initials] June 11, 1997  
Speed: 90 RPM M June 11, 1997 [initials] June 11, 1997  
M June 11, 1997

4. Weight of tablets Gal 205 Bal.

[See original for numbers.]

[illegible initials and date]

FOURNIER 1001737



5. Reading  
photospectrometer Gal 233 chronometer Gal 124  
2 mm tank - 290nm

M 6/11/97

LF 178 Ter LOT 458 CO194, 0.01M 90 RPM

Lambda. No. Value\_E

[See original for numbers.]

[illegible initials and date]

FOURNIER 1001738

036

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	LF178 Ter Tablets Lot CO194 - 0.1M 90 tprm
DATE	06/11/97 RC	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 33 RC
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.01 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001739

June 12, 1997

Dissolution LF 178 Ter Tablets

Lot CO197 – 160 mg

LSNa: 0.1M – 90 RPM

1. Preparation of the dissolution medium Gal 065 Balance

Purified water

[See original for numbers.]

Tare Total weight of water

Net Weight of LSNa for a 0.1M medium

Gross

2. Weight of the Dissolution Bowls Gal 065 Bal  
1 liter of 0.1 M LSNa weighs 1004.0 g

M July 10, 1997

FOURNIER 1001740

038

[See original for numbers.]

Tare

Net

Gross

3. Dissolution conditions  
Dissolutest Gal 091 Sotax AT 7

Temp.=37°C ±0.5°C RC June 12, 1997 Ab June 12, 1997  
Speed: 90 RPM RC June 12, 1997 Ab June 12, 1997

4. Weight of tablets Gal 205 Balance

[See original for numbers.]

[illegible initials and date]

FOURNIER 1001741

5. Reading  
on photospectrometer KONTRON 922 Gal 233  
2 mm optical view tanks – 290 nm

FIXED WAVELENGTH 290 NM 2 MM LF 178 Ter LOT CO197, 0.1 NM 90 RPM  
LSNA

Lambda.	No.	Value_E
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[See original for numbers.]

KONTRON INSTRUMENTS UVIKON 922	Operator	RC
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FOURNIER 1001742

040

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	LF178 Ter Tablets Lot CO 197
DATE	06/12/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 37
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.1 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001743

June 12, 1997

Dissolution LF 178 Ter Tablets

Lot CO200 – 160 mg

LSNa: 0.1M – 90 RPM

1. Preparation of the dissolution medium Gal 065 Balance

Purified water

[See original for numbers.]

Tare Total weight of water

Net Weight of LSNa for a 0.1M medium

Gross

2. Weight of the Dissolution Bowls Gal 065 Bal

1 liter of 0.1 M LSNa weighs 1004.09 g

FOURNIER 1001744

042

[See original for numbers.]

Tare  
Net  
Gross

3. Dissolution conditions  
Dissolutest Gal 091 Sotax AT 7

Temp.=37°C ±0.5°C RC June 13, 1997 Ab June 13, 1997  
Speed: 90 RPM RC June 13, 1997 Ab June 13, 1997

4. Weight of tablets Gal 205 Balance

[See original for numbers.]

FOURNIER 1001745

Highly Confidential Subject to Protective Order



5. Reading  
on photospectrometer UVIKON 922-Gal 233  
2 mm optical view tanks – 290 nm

FIXED WAVELENGTH 290 NM 2 MM LSNA 0.1M 90 RPM LOT CO200 LF178 Ter

Lambda.	No.	Value_E
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[See original for numbers.]

KONTRON INSTRUMENTS UVIKON 922

Operator

RC

FOURNIER 1001746

044

DISSOLUTION

[See original for file name.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for names and numbers.]	TITLE	LF178 Ter Tablets Lot CO200 0.1M 90 RPM
DATE	06/13/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 41
APPARATUS		FILE	[See original for file name.]
WAVELENGTH		ELUANT	0.1 M LSNa
TANK in mm		AGITATION	

SAMPLE PREPARATION

theoretical mass	[See original for numbers.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for numbers.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for numbers.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[illegible initials and date]

FOURNIER 1001747

045

Parallel use of the 7<sup>th</sup> bowl for readings with or without Millex HA filtration (ref. SLHA025NB)  
of 0.45  $\mu$ m

Removal 10 ml, 5 filtered ml immediately and 5 left in the syringe and reading after 20 min  
[illegible] of time minimum

[See original for figures.]

Tare

Net

Gross

RC

RC

FIXED WAVELENGTH 290 NM 2MM LSNA 0.1M 90TPM LOT C0200 LF178TER WITH  
6/13/1997 11:42 am

Lambda	No.	Value_E
[See original for figures.]		

KONTRON INSTRUMENTS UVIKON 922      Operator RC

FIXED WAVELENGTH 290NM 2MM LSNA 0.1M 90TPM LOT C0200 LF178TER  
WITHOUT

6/13/1997 11:47 am

Lambda	No.	Value_E
[See original for figures.]		

M 7/10/1997

KONTRON INSTRUMENTS UVIKON 922      Operator RC

FOURNIER 1001748

Highly Confidential Subject to Protective Order

046

DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets PK CO200 – 0.1 M 90tpm
DATE	06/13/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 45
APPARATUS	[See original for text in English.]	FILE	[See original for text in English.]
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.1 M
TANK in mm	[See original for text in English.]	AGITATION	90 TPM WITH FILTRATION

SAMPLE PREPARATION

theoretical mass	[See original for figures.]
theoretical dose	

in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion	[See original for figures.]					
quantity of active ingredient						

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1
[See original for figures.]		

Time

*Difference between the two readings in % dissolved*

[See original for figures.]

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1
[See original for figures.]		

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1
[See original for figures.]		

FOURNIER 1001749

DISSOLUTION

{See original for text in English.}  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	{See original for text in English.}	TITLE	LF178TER Tablets PK CO200 – 0.1 M 90tpm
DATE	06/13/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 45
APPARATUS	{See original for text in English.}	FILE	{See original for text in English.}
WAVELENGTH	{See original for text in English.}	ELUANT	LSNa 0.1 M
TANK in mm	{See original for text in English.}	AGITATION	90 TPM WITH FILTRATION

SAMPLE PREPARATION

theoretical mass	{See original for figures.}
theoretical dose	

in mg

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
{See original for figures.}					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1
{See original for figures.}		

*Given the difference between the two measurements, it would be better to do the 0.45 pm filtration just after removal and not before the reading. In any event, the filtration has to be adapted according to the withdrawal times.*

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1
{See original for figures.}		

*1<sup>st</sup> case: reading at times 20, 40, 60, and 120 m, remove, put in a 0.45 pm Millex filter, take the time interval between the two readings is sufficient to take readings after removal*

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1
{See original for figures.}		

*2<sup>nd</sup> case: readings at times 5, 10, 15, 20, and 30 minutes, remove, put in a 0.45 pm Millex filter in a test tube, waiting for the reading.*

M 7/10/1997

FOURNIER 1001750

048

June 17, 1997

Dissolution of LF178Ter Tablets

Lot 158 – 100 mg

LSNa 0.1 M – 90 TPM

1. Preparation of the Dissolution Medium

Balance GAL 065

[See original for figures.]

*Total of purified water weighed*

[See original for equation.]

*Tare*

*Tare*

*Net*

*Net*

*Gross*

*Gross*

*Tare*

*Tare*

*Net*

*Net*

*Gross*

*Gross*

*Tare*

*Weight of LSNa in a 0.1M medium*

*Tare*

[See original for equation.]

*Net*

*Net*

*Gross*

*Gross*

2. Weight of Dissolution Bowls

Balance GAL 065

1 liter of LSNa 0.1 M weighs 1004.0 g

M 7/10/1997

FOURNIER 1001751

[See original for figures.]

049

<i>Tare</i>	<i>Tare</i>
<i>Net</i>	<i>Net</i>
<i>Gross</i>	<i>Gross</i>
<i>Tare</i>	<i>Tare</i>
<i>Net</i>	<i>Net</i>
<i>Gross</i>	<i>Gross</i>

3. Dissolution Conditions  
Dissolutest GAL 091-Sotax AT7

Temperature: $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$	RC June 17, 1997	Ab June 17, 1997
Speed: 90 TPM	RC June 17, 1997	Ab June 17, 1997

4. Weight of Tablets      Balance GAL205

[See original for figures.]

M 7/10/1997

FOURNIER 1001752

Highly Confidential Subject to Protective Order

050

5. Readings - on photospectrometer UVIKON 922 Gal 233  
2 mm tanks to 290 nm.

FIXED WAVELENGTH 290 NM 2 MM LF178TER LOT 158, LSNA 0.1M 90 TPM

6/17/1997 4:16 pm

Lambda.	No.	Value_E
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[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922

Operator

RC

M 7/10/1997

FOURNIER 1001753

Highly Confidential Subject to Protective Order



DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets Lot 158 – 0.1 M 90 tpm s
DATE	06/17/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 48
APPARATUS	[See original for text in English.]	FILE	M:\commun\ginq\lf178ter\dissolution\lot PK 158 0.1 90 tpm
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.1 M
TANK in mm	[See original for text in English.]	AGITATION	[See original for text in English.]

SAMPLE PREPARATION

theoretical mass	[See original for figures.]
theoretical dose	

in mg

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

[See original for graphs.]

% dissolved

quantity dissolved

time

time

RC

FOURNIER 1001754

June 17, 1997

Dissolution of LF178Ter Tablets

Lot 234 – 100 mg

LSNa 0.1 M – 90 TPM

1. Preparation of the Dissolution Medium

See page 048.

2. Weight of Dissolution Bowls Balance GAL 065

1 liter of LSNa 0.1M weighs 1004.0 g

[See original for figures.]

Tare

Tare

Tare

Net

Net

Net

Gross

Gross

Gross

Tare

Tare

Tare

Net

Net

Net

Gross

Gross

Gross

RC

3. Dissolution Conditions

Dissolutest Sotax AT7 – GAL 091

Temperature: 37°C ± 0.5°C RC June 18, 1997 CC June 18, 1997

Speed: 90 TPM RC June 18, 1997 CC June 18, 1997

4. Weight of Tablets Balance GAL205

M 7/10/1997

FOURNIER 1001755

[See original for figures.]

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tanks with optical trajectory of 290 nm

FIXED WAVELENGTH 290 NM 2 MM LF178TER LOT 234, LSNA 0.1M 90 TPM  
6/18/1997 11:23 am

Lambda.	No.	Value_E
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[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922      Operator      RC

FOURNIER 1001756

054

DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets PK Lot 234 – 0.1 M 90rpm
DATE	06/18/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 52
APPARATUS	[See original for text in English.]	FILE	[See original for text in English.]
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.1 M
TANK in mm	[See original for text in English.]	AGITATION	[See original for text in English.]

SAMPLE PREPARATION

theoretical mass	[See original for figures.]
theoretical dose	

in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/lDISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

[See original for graphs.]

% dissolved

quantity dissolved

time

time

RC

M 7/10/1997

FOURNIER 1001757

Highly Confidential Subject to Protective Order

June 18, 1997

055

Dissolution of LF178Ter Tablets

Lot 234 – 100 mg

LSNa 0.05 M – 120 TPM

1. Preparation of the Dissolution Medium - Balance GAL 065

[See original for figures.]

Total of purified water weighed  
[See original for equation.]

Tare

Tare

Net

Net

Gross

Gross

Tare

Tare

Net

Net

Gross

Gross

Tare

Weight of LSNa in a 0.05M medium  
[See original for equation.]

Net

Gross

2. Weight of Dissolution Bowls Balance GAL 065  
1 liter of LSNa 0.05 M weighs 1002.0 g

M 7/10/1997

FOURNIER 1001758

[See original for figures.]

<i>Tare</i>	<i>Tare</i>	<i>Tare</i>
<i>Net</i>	<i>Net</i>	<i>Net</i>
<i>Gross</i>	<i>Gross</i>	<i>Gross</i>
<i>Tare</i>	<i>Tare</i>	<i>Tare</i>
<i>Net</i>	<i>Net</i>	<i>Net</i>
<i>Gross</i>	<i>Gross</i>	<i>Gross</i>

3. Dissolution Conditions  
*Dissolutest Sotax AT7 – GAL 091*

<i>Temperature: 37°C ± 0.5°C</i>	<i>RC June 18, 1997</i>	<i>CC June 18, 1997</i>
<i>Speed: 120 TPM</i>	<i>RC June 18, 1997</i>	<i>CC June 18, 1997</i>

4. Weight of Tablets      *Balance GAL205*

[See original for figures.]

5. Readings  
*on photospectrometer UVIKON 922 Gal 233*  
*2 mm tanks with optical trajectory of 290 nm*

M 7/10/1997

FOURNIER 1001759

057

FIXED WAVELENGTH 290 NM 2 MM LF178TER LOT 234, LSNA 0.05M 120 TPM  
6/18/1997 4:40 pm

Lambda.	No.	Value_E
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[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922	Operator	RC
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FOURNIER 1001760

Highly Confidential Subject to Protective Order

058

DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets PK Lot 234 – 0.05 M 120 tpm
DATE	06/18/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, page 55
APPARATUS	[See original for text in English.]	FILE	[See original for text in English.]
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.05 M
TANK in mm	[See original for text in English.]	AGITATION	[See original for text in English.]

SAMPLE PREPARATION

theoretical mass	[See original for figures.]
theoretical dose	

in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

[See original for graphs.]

% dissolved

quantity dissolved

time

time

M 7/10/1997

FOURNIER 1001761

Highly Confidential Subject to Protective Order



June 18, 1997

Dissolution of LF178Ter Tablets

Lot CO194 – 160 mg

LSNa 0.05 M – 120 TPM

1. Preparation of the Dissolution Medium

See page 055.

2. Weight of Dissolution Bowls

RC  
Balance GAL 205 065

1 liter of 0.05M LSNa weighs 1002.0 g

Purified water

[See original for figures.]

Tare	Tare	Tare
Net	Net	Net
Gross	Gross	Gross
Tare	Tare	Tare
Net	Net	Net
Gross	Gross	Gross

3. Dissolution Conditions

Dissolutest Sotax AT7 – GAL 091

Temperature: 37°C ± 0.5°C	RC June 19, 1997	[initials] June 19, 1997
Speed: 120 TPM	RC June 19, 1997	[initials] June 19, 1997

M 7/10/1997

FOURNIER 1001762

060

4. Weight of Tablets      *Balance GAL205*

[See original for figures.]

5. Readings  
*on photospectrometer UVIKON 922 Gal 233*  
*2 mm tanks with optical trajectory of 290 nm*

FIXED WAVELENGTH 290 NM 2 MM LF178TER LOT CO194, LSNA 0.05M 120 TPM  
6/19/1997 11:10 am

Lambda.	No.	Value_E
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[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922      Operator      RC

*M 7/10/1997*

FOURNIER 1001763

DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets Lot C0194 – 0.05 M 120tpm
DATE	06/19/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 59
APPARATUS	[See original for text in English.]	FILE	[See original for text in English.]
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.05 M
TANK in mm	[See original for text in English.]	AGITATION	[See original for text in English.]

SAMPLE PREPARATION

theoretical mass	[See original for figures.]
theoretical dose	

in mg

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion	[See original for figures.]					
quantity of active ingredient						

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

[See original for graphs.]

% dissolved

quantity dissolved

time

time

RC

FOURNIER 1001764

062

June 19, 1997

Dissolution of LF178Ter Tablets

Lot CO197 – 160 mg

LSNa 0.05 M – 120 TPM

1. Preparation of the Dissolution Medium Balance GAL 065

[See original for figures.]

purified water

*Tare*

*Tare*

Total weight of purified water

[See original for equation.]

*Net*

*Net*

*Gross*

*Gross*

*Tare*

*Tare*

*Net*

*Net*

*Gross*

*Gross*

*Tare*

*Weight of LSNa for a 0.05 M medium*

[See original for equation.]

*Net*

*Gross*

*Tare*

*Net*

*Gross*

2. Weight of Dissolution Bowls

Balance GAL 065

1 liter of 0.05M LSNa weighs 1002.0 g

FOURNIER 1001765

<i>Tare</i>	<i>Tare</i>	<i>Tare</i>
<i>Net</i>	<i>Net</i>	<i>Net</i>
<i>Gross</i>	<i>Gross</i>	<i>Gross</i>
<i>Tare</i>	<i>Tare</i>	<i>Tare</i>
<i>Net</i>	<i>Net</i>	<i>Net</i>
<i>Gross</i>	<i>Gross</i>	<i>Gross</i>

3. Dissolution Conditions  
*Dissolutest Sotax AT7 – GAL 091*

<i>Temperature: 37°C ± 0.5°C</i>	<i>June 19, 1997</i>	<i>Ab June 19, 1997</i>
<i>Speed: 120 TPM</i>	<i>RC June 19, 1997</i>	<i>Ab June 19, 1997</i>

4. Weight of Tablets - *Balance GAL205*

[See original for figures.]

5. Readings  
*on photospectrometer UVIKON 922 Gal 233*  
*2 mm tanks with optical trajectory of 290 nm*

M 7/10/1997

FOURNIER 1001766

064

FIXED WAVELENGTH 290 NM 2 MM LF178TER LOT C0194, LSNA 0.05M 120 TPM  
6/19/1997 4:22 pm

Lambda.	No.	Value_E
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[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922	Operator	RC
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FOURNIER 1001767

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets Lot CO197 – 0.05 M 120tpm
DATE	06/19/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 62
APPARATUS	[See original for text in English.]	FILE	[See original for text in English.]
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.05 M
TANK in mm	[See original for text in English.]	AGITATION	[See original for text in English.]

SAMPLE PREPARATION

theoretical mass	[See original for figures.]
theoretical dose	

in mg

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

[See original for graphs.]

% dissolved

quantity dissolved

time

time

FOURNIER 1001768

066

June 19, 1997

Dissolution of LF178Ter Tablets

Lot C0200 – 160 mg

LSNa 0.05 M – 120 TPM

1. Preparation of the Dissolution Medium

See page 062.

2. Weight of Dissolution Bowls Balance GAL 065

1 liter of 0.05M LSNa weighs 1002.0 g

[See original for figures.]

Tare

Tare

Tare

Net

Net

Net

Gross

Gross

Gross

Tare

Tare

Tare

Net

Net

Net

Gross

Gross

Gross

3. Dissolution Conditions

Dissolutest Sotax AT7 – GAL 091

Temperature: 37°C ± 0.5°C RC June 20, 1997

[initials] June 20, 1997

Speed: 120 TPM

RC June 20, 1997

[initials] June 20, 1997

FOURNIER 1001769

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4. Weight of Tablets      Balance GAL205

[See original for figures.]

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tanks with optical trajectory of 290 nm

290 NM 2 MM LF178TER LOT C0200, 0.05M 120 TPM

6/20/1997 10:58 am

Lambda.      No.      Value\_E

[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922      Operator      RC

M 7/10/1997

FOURNIER 1001770

068

DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets Lot C0200 – 0.05 M 120tpm
DATE	06/20/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 66
APPARATUS	[See original for text in English.]	FILE	[See original for text in English.]
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.05 M
TANK in mm	[See original for text in English.]	AGITATION	[See original for text in English.]

SAMPLE PREPARATION

theoretical mass	[See original for figures.]
theoretical dose	

in mg

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

[See original for graphs.]

% dissolved

quantity dissolved

time

time

FOURNIER 1001771

Highly Confidential Subject to Protective Order

*Lot 234 – 100 mg*

LSNa 0.025 M – 75 TPM

### 1. Preparation of the Dissolution Medium

*Balance GAL 205 065*  
*Total weight of purified water*  
 [See original for equation.]

[See original for figures.]

*Tare*

*Tare*

*Net.*

*Net*

*Gross*

*Gross*

*Tare*

*Weight of 0.025M LSNa Medium*  
[See original for equation.]

*Net*

*Tare*

*Gross*

*Net*

*Gross*

 $RC$ 

2. Weight of Dissolution Bowls Balance GAL 065  
1 liter of 0.025M LSNa weighs 1001.0 g

### *Tare, Net, Gross*

### *Tare, Net, Gross*

### *Tare, Net, Gross*

[See original for figures.]

*M 7/10/1997*

FOURNIER 1001772

070

*Tare, Net, Gross*

*Tare, Net, Gross*

*Tare, Net, Gross*

[See original for figures.]

3. *Dissolution Conditions*

*Dissolutest Sotax AT7 – GAL 091*

*Temperature: 37°C ± 0.5°C    RC June 20, 1997    June 20, 1997 Ab*

*Speed: 90 75 TPM                  RC June 20, 1997    June 20, 1997 Ab*

*RC*

4. *Weight of Tablets*      *Balance GAL205*

[See original for figures.]

5. *Readings*

*on photospectrometer UVIKON 922 Gal 233*

*2 mm tanks with optical trajectory of 290 nm*

FOURNIER 1001773

071

FIXED WAVELENGTH 290 NM 2 MM LF178TER LOT 234, LSNA 0.025M 75 TPM

6/20/1997 4:39 pm

Lambda.	No.	Value_E
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[See original for figures.]

*M 7/10/1997*

FOURNIER 1001774

Highly Confidential Subject to Protective Order

072  
DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets Lot PK 234 – 0.025 M 75tpm
DATE	06/20/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 69
APPARATUS	[See original for text in English.]	FILE	[See original for text in English.]
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.025 M
TANK in mm	[See original for text in English.]	AGITATION	[See original for text in English.]

SAMPLE PREPARATION

theoretical mass	[See original for figures.]	in mg
theoretical dose		

mass of test portion quantity of active ingredient	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
	[See original for figures.]					

Control 100  
mg/l

--

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

[See original for graphs.]

% dissolved

time

quantity dissolved

time

FOURNIER 1001775

Highly Confidential Subject to Protective Order

June 24, 1997

Dissolution of LF178Ter TabletsLot 158 - 100 mg x 2 = 200 mgLSNa 0.025 M - 75 TPM1. Preparation of the Dissolution Medium

Purified Water

Balance GAL065

Total weight of purified water

[See original for equation.]

[See original for figures.]

Weight of 0.025M LSNa Medium

[See original for equation.]

Tare

Net

Gross

Tare

Net

Gross

Tare

Net

Gross

Tare

Net

Gross

2. Weight of Dissolution Bowls

Balance GAL 065

1 liter of 0.025M LSNa weighs 1001.0 g

M 7/10/1997

FOURNIER 1001776

[See original for figures.]

<i>Tare</i>	<i>Tare</i>	<i>Tare</i>
<i>Net</i>	<i>Net</i>	<i>Net</i>
<i>Gross</i>	<i>Gross</i>	<i>Gross</i>
<i>Tare</i>	<i>Tare</i>	<i>Tare</i>
<i>Net</i>	<i>Net</i>	<i>Net</i>
<i>Gross</i>	<i>Gross</i>	<i>Gross</i>

3. Dissolution Conditions

*Dissolutest Sotax AT7 – GAL 091*

*Temperature: 37°C ±0.5°C [initials] June 24, 1997*

*RC June 24, 1997*

*Speed: 75 TPM*

*[initials] June 24, 1997*

*RC June 24, 1997*

4. Weight of Tablets      *Balance GAL205*

[See original for figures.]

5. Readings

*on photospectrometer UVIKON 922 Gal 233*

*2 mm tanks with optical trajectory of 290 nm*

*M 7/10/1997*

FOURNIER 1001777



075  
FIXED WAVELENGTH 290 NM 2 MM LF178TER 2 CPR LOT 158, LSNA 0.025M 75 TPM  
6/24/1997 5:38 pm

Lambda. No. Value\_E

[See original for figures.]

*M 7/10/1997*

FOURNIER 1001778

Highly Confidential Subject to Protective Order

076  
DISSOLUTION

[See original for text in English.]  
version dated: February 6, 1997

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	[See original for text in English.]	TITLE	LF178TER Tablets Lot PK 158 – 0.025 M 75tpm 2 tablets
DATE	06/24/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 73
APPARATUS	[See original for text in English.]	FILE	M:\commun\ginqlf178ter\dissolution\lot PK 158 0.025 75 tpm 2 tablets
WAVELENGTH	[See original for text in English.]	ELUANT	LSNa 0.025 M
TANK in mm	[See original for text in English.]	AGITATION	[See original for text in English.]

SAMPLE PREPARATION

theoretical mass	[See original for figures.]
theoretical dose	

in mg

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

[See original for graphs.]

% dissolved

quantity dissolved

time

time

FOURNIER 1001779

Highly Confidential Subject to Protective Order

6/24/97

Dissolution of LF178Ter TabletsLot 2398/01 RG -14 kgLSNa 0.05 M - 120 TPM

1. Preparation of the Dissolution Medium  
Purified Water

Balance GAL065  
Total weight of purified water

[See original for figures.]

Tare  
Net  
Gross

Weight of 0.05M LSNa Medium  
[See original for figures.]

2. Weight of Dissolution Bowls Balance GAL 265  
1 liter of 0.05M LSNa weighs 1002.0 g

[initials] 7/10/97

FOURNIER 1001780

078  
Tare  
Net  
Gross

[See original for figures.]

3. Dissolution Conditions  
Dissolutest Sotax AT7 -- GAL 091

Temperature: 37°C ± 0.5°C	[initials] 6/25/97	[initials] 6/25/97
Speed: 120 TPM	[initials] 6/25/97	[initials] 6/25/97

4. Weight of Tablets      Balance GAL205

[See original for figures.]

[initials] 7/10/97  
FOURNIER 1001781

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tanks with optical trajectory of 290 nm

FIXED WAVELENGTH 290 NM 2 MM LF 178TER 2398/01RG 14 KG LSNA 0.05M  
120 TPM

Lambda.      No.      Value\_E

[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922

Operator      [initials]

[initials] 7/10/97

FOURNIER 1001782

080

DISSOLUTION[See original for file name.]  
version dated: 2/6/97CONDITIONS AND OPERATING PROCEDURES

OPERATOR	ROSSELIN C	TITLE	LF178TER Tablets Lot 2398/01 RG 14kg- 0.05 M 120 tpm
DATE	06/25/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 077
APPARATUS	GAL091 GAL 233	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.05
TANK in mm	2 mm	AGITATION	120 tpm

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/10/97  
FOURNIER 1001783

Highly Confidential Subject to Protective Order

6/25/97

Dissolution of LF178Ter TabletsLot 2401/01 RG – 120 kgLSNa 0.05 M – 120 TPM1. Preparation of the Medium

See page 077.

2. Weight of Dissolution Bowls Balance GAL 065

1 liter of 0.05M LSNa weighs 1002.0 g

[See original for figures.]

Tare

Net

Gross

3. Dissolution Conditions

Dissolutest Sotax AT7 – GAL 091

Temperature:  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  [initials] 6/25/97 [initials] 6/25/97

Speed: 120 TPM [initials] 6/25/97 [initials] 6/25/97

[initials] 7/10/97

FOURNIER 1001784

4. Weight of Tablets Balance GAL205

[See original for figures.]

FIXED WAVELENGTH 290 NM 2 MM LF 178TER 2401/01RG 20 KG LSNA 0.05M  
120 TPM

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tanks with optical trajectory of 290 nm

Lambda	No.	Value_E
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[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922

Operator [initials]

[initials] 7/10/97  
FOURNIER 1001785

Highly Confidential Subject to Protective Order



DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	ROSSELIN C	TITLE	LF178TER Tablets Lot 2401/01 RG 20kg- 0.05 M 120 tpm
DATE	06/25/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 081
APPARATUS	GAL091 GAL233	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.05M
TANK in mm	2 mm	AGITATION	120 tpm

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

[initials] 7/10/97

quantity dissolved

[See original for graph.]

time

FOURNIER 1001786

Highly Confidential Subject to Protective Order

6/27/97

Dissolution of LF178Ter Tablets

Lot 2401/01 RG – 15 kg

LSNa 0.05 M – 120 TPM

- |    |  |                                |
|----|--|--------------------------------|
| 1. | <u>Preparation of the Dissolution Medium</u> | Balance GAL 065                |
|    | Purified Water                               | Total Weight of Purified Water |

[See original for figures.]

[See original for figures.]

Tare

Net

Gross

Weight of LSNA to have a 0.05M medium

[See original for figures.]

- |    |                                       |                 |
|----|---------------------------------------|-----------------|
| 2. | <u>Weight of Dissolution Bowls</u>    | Balance GAL 065 |
|    | 1 liter of 0.05M LSNa weighs 1002.0 g |                 |

[initials] 7/10/97  
FOURNIER 1001787

Highly Confidential Subject to Protective Order

[See original for figures.]

Tare  
Net  
Gross

3. Dissolution Conditions  
Dissolutest AT7 Sotax GAL 091

Temperature: $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$	[initials] 6/27/97	[initials] 6/27/97
Speed: 120 TPM	[initials] 6/27/97	[initials] 6/27/97

[See original for figures.]

[initials] 7/10/97

FOURNIER 1001788

Highly Confidential Subject to Protective Order

086

FIXED WAVELENGTH 290 NM 2 MM LF 178TER 2401/01RG 15 KG LSNA 0.05M  
120 TPM

Lambda	No.	Value_E
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[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922

Operator [initials]

[initials] 7/10/97  
FOURNIER 1001789

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	ROSSELIN C	TITLE	LF178TER Tablets Lot 2401/01 RG 15kg- 0.05 M 120 tpm
DATE	06/27/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 084
APPARATUS	GAL 091 GAL 233	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.05
TANK in mm	2 mm	AGITATION	120 tpm

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	Volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/10/97  
FOURNIER 1001790

088

Dissolution of LF178Ter Tablets

Lot 2398/01 RG – 14 kg

LSNa 0.1 M – 90 TPM

1. Preparation of the Dissolution Medium Balance GAL 065

Purified Water

Total Weight of Purified Water

[See original for figures.]

[See original for figures.]

Tare  
Net  
Gross

Weight of LSNA to have a 0.1M medium  
[See original for figures.]

2. Weight of Dissolution Bowls Balance GAL 065  
1 liter of 0.1M LSNa weighs 1004.0 g

[initials] 7/10/97  
FOURNIER 1001791

Highly Confidential Subject to Protective Order

[See original for figures.]

Tare  
Net  
Gross

3. Dissolution Conditions  
Dissolutest Sotax AT7 GAL 091

Temperature: $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$	[initials] 7/1/97	[initials] 7/1/97
Speed: 90 TPM	[initials] 7/1/97	[initials] 7/1/97

4. Weight of Tablets      Balance GAL 205

[See original for figures.]

[initials] 7/10/97

FOURNIER 1001792

090

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tanks with optical trajectory of 290 nm

FIXED WAVELENGTH 290 NM 2 MM LF 178TER 2398/01RG 14 KG LSNA 0.1M 90  
TPM

Lambda      No.      Value\_E

[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922

Operator      [initials]

[initials] 7/10/97

FOURNIER 1001793

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DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	ROSSELIN C	TITLE	LF178TER Tablets Lot 2398/01 RG 14kg- 0.1 M 90 tpm
DATE	07/01/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 088
APPARATUS	GAL 091 GAL233	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.1M
TANK in mm	2 mm	AGITATION	90 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	Volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/10/97  
FOURNIER 1001794

092  
7/1/97

Dissolution of LF178Ter Tablets  
Lot 2401/01 RG – 15 kg  
LSNa 0.1 M – 90 TPM

1. Preparation of the Dissolution Medium  
See page 88.
2. Weight of Dissolution Bowls      Balance GAL 065  
1 liter of 0.1M LSNa weighs 1004.0 g

[See original for figures.]

Tare  
Net  
Gross

3. Dissolution Conditions  
Dissolutest Sotax AT7 GAL 091

Temperature: 37°C ± 0.5°C	[initials] 7/1/97	[initials] 7/1/97
Speed: 90 TPM	[initials] 7/1/97	[initials] 7/1/97

[initials] 7/10/97

FOURNIER 1001795

4. Weight of Tablets Balance GAL 205

[See original for figures.]

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tanks with optical trajectory of 290 nm

FIXED WAVELENGTH 290 NM 2 MM LF 178TER 2401/01RG 15 KG LSNA 0.1M 90  
TPM

Lambda	No.	Value_E
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[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922  
[initials] 7/10/97

Operator [initials]

FOURNIER 1001796

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	ROSSELIN C	TITLE	LF178TER Tablets Lot 2401/01 RG 15kg- 0.1 M 90 tpm
DATE	07/01/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 092
APPARATUS	GAL091 GAL233	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.1M
TANK in mm	2 mm	AGITATION	90 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	Volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001797

7/1/97

095

Dissolution LF178TER Tablets  
Lot 2401/01 RG – 20 kg  
LSNA 0.1M – 90 TPM

1. <u>Preparation of the Dissolution Medium</u>	Balance GAL 065
Purified Water	Total Weight of Purified Water
[See original for figures.]	[See original for figures.]
Tare	
Net	Weight of LSNa for a medium of 0.1M
Gross	[see original for details]

2. <u>Weight of Dissolution Bowls</u>	Balance GAL 065
1 liter of LSNa 0.1M weighs 1004.0g	

[initials] 7/10/97

FOURNIER 1001798

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096

[See original for figures.]

Tare

Net

Gross

7/2/97

3. Dissolution Conditions

Dissolutest Sotax AT7-GAL 091

Temperature: 37°C±0.5°C [initials] 7/2/97 [initials] 7/2/97

Speed: 90 TPM [initials] 7/2/97 [initials] 7/2/97

4. Weight of Tablets GAL 205

[See original for figures.]

[initials] 7/10/97  
FOURNIER 1001799

Highly Confidential Subject to Protective Order

5. Readings

photospectrometer UVIKON 922 GAL 233 in 2mm tanks with an optical trajectory of 290 nm

FIXED WAVELENGTH 290 NM 2 MM LF178TER 2401/01RG 20 KG 0.1 TPM

Lambda	No.	Value_E
--------	-----	---------

KONTRON INSTRUMENTS UVIKON 922

Operator [initials]

[initials] 7/10/97

FOURNIER 1001800

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098

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	ROSSELIN C	TITLE	LF178TER Tabletslot2401/01
DATE	07/02/97	NOTEBOOK NO.	RG 20kg - 0.1M
APPARATUS	GAL091 GAL233	FILE	LF 178 Ter Dissolution no. 2,
WAVELENGTH	290 nm	ELUANT	p. 095
TANK in mm	2 mm	AGITATION	[See original for file name.]
			LSNa 0.1M
			90 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/10/97  
FOURNIER 1001801

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7/3/97

099

Dissolution of LF178Ter Tablets

Lot 2395/01 RG – 14 kg

LSNa 0.1 M – 90 TPM

- |    |  |                                  |
|----|--|----------------------------------|
| 1. | <u>Preparation of the Dissolution Medium</u> | Balance GAL 065                  |
|    | Purified Water                               | Total Weight of Purified Water   |
|    | [See original for figures.]                  | [See original for figures.]      |
|    | Tare   |                                  |
|    | Net  | Weight of LSNA for a 0.1M medium |
|    | Gross  | [See original for figures.]      |

- |    |                                      |                 |
|----|--------------------------------------|-----------------|
| 2. | <u>Weight of Dissolution Bowls</u>   | Balance GAL 065 |
|    | 1 liter of 0.1M LSNa weighs 1004.0 g |                 |

[initials] 7/10/97

FOURNIER 1001802

Highly Confidential Subject to Protective Order

100

[See original for figures.]

Tare

Net

Gross

3. Dissolution Conditions  
Dissolutest Sotax AT7 GAL 091

Temperature: 37°C ± 0.5°C	[initials] 7/3/97	[initials] 7/3/97
Speed: 90 TPM	[initials] 7/3/97	[initials] 7/3/97

4. Weight of Tablets Balance GAL 205

[See original for figures.]

[initials] 7/10/97

FOURNIER 1001803

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tanks with optical trajectory of 290 nm

FIXED WAVELENGTH 290 NM 2 MM LF 178TER 2395/01RG 14 KG LSNA 0.1M 90  
TPM

Lambda      No.      Value\_E

[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922      Operator      [initials]

[initials] 7/10/97

FOURNIER 1001804

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	ROSSELIN C	TITLE	LF178TER Tablets Lot 2395/01 RG 14kg- 0.1 M 90 tpm
DATE	07/03/97	NOTEBOOK NO.	LF 178 Ter Dissolution no. 2, p. 099
APPARATUS	GAL 091 GAL 233	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.1M
TANK in mm	2 mm	AGITATION	90 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	Volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/10/97  
FOURNIER 1001805

7/3/97

103

Dissolution of LF178 Tablets

Lot 2395/01 RG – 14 kg

LSNa 0.05 M – 120 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065  
Purified Water      Total Weight of Purified Water  
[See original for figures.]      [See original for figures.]  
Tare  
Net      Weight of LSNA for a 0.05M medium  
Gross      [See original for figures.]

2. Weight of Dissolution Bowls      Balance GAL 065  
1 liter of 0.05M LSNa weighs 1002.0 g

[initials] 7/10/97

FOURNIER 1001806

Highly Confidential Subject to Protective Order

104

[See original for figures.]

Tare

Net

Gross

3. Dissolution Conditions  
Dissolutest Sotax AT7 GAL 091

Temperature: $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$	[initials] 7/3/97	[initials] 7/3/97
Speed: 120 TPM	[initials] 7/3/97	[initials] 7/3/97

4. Weight of Tablets Balance GAL 205

[See original for figures.]

[initials] 7/10/97

FOURNIER 1001807

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tanks with optical trajectory of 290 nm

FIXED WAVELENGTH 290 NM 2 MM LF 178TER 2395/01RG 14 KG LSNA 0.05M  
120 TPM

Lambda      No.      Value\_E

[See original for figures.]

KONTRON INSTRUMENTS UVIKON 922      Operator      [initials]

No sample taken at 20 minutes; dissolution needs to be redone.

7/4/97

1. Preparation of Dissolution Medium Balance GAL 065

[initials] 7/10/97

FOURNIER 1001808

106  
7/8/97

LF178Ter Tablets  
Lot 2395/01 RG – 14 kg  
LSNa 0.05 M – 120 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065

Weight of Water

WATER

Total Weight of Water

Tare  
Net  
Gross

[See original for figures.]

Weight of LSNA for a 0.05M medium  
[See original for figures.]

[initials] 7/9/97

FOURNIER 1001809



2. Weight of Dissolution Bowls Balance GAL 065  
1 liter of 0.05M LSNa weighs 1002.0 g

[See original for figures.]

Tare  
Net  
Gross

3. Dissolution Conditions  
Dissolutest Sotax AT7 GAL 091

Temperature: 37°C ± 0.5°C	[initials] 7/8/97	[initials] 7/8/97
Speed: 120 TPM	[initials] 7/3/97	[initials] 7/8/97

4. Weight of Tablets Balance GAL 205

[See original for figures.]

[initials] 7/9/97

FOURNIER 1001810

108

5. Readings  
on photospectrometer UVIKON 922 Gal 233  
2 mm tank – reading at 290 nm

LF 178 TER LOT RG 2395/01 14KG LSNA 0.05M 120 TPM

Lambda      No.      Value\_E

[See original for figures and text in English.]

[initials] 7/9/97

FOURNIER 1001811

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DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF178TER RG 2395/01 14kg
DATE	07/08/97	NOTEBOOK NO.	LF 178 Ter No. 2, p. 106
APPARATUS	GAL 233 091	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.05 M
TANK in mm	2	AGITATION	120 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	Volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials]

FOURNIER 1001812

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7/9/97  
110

Dissolution of LF178Ter Tablets  
Lot RG 2395/01 – 14 kg  
LSNa 0.025 M – 75 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065

Weight of Water

WATER

Amount of Water Weighed

[See original for figures.]

[See original for figures.]

Tare  
Net  
Gross

Weight of LSNA for a 0.025M medium  
[See original for figures.]

[initials] 7/9/97

FOURNIER 1001813

Highly Confidential Subject to Protective Order

2. Weight of Dissolution Bowls Balance GAL 065  
1 liter of 0.025M LSNa weighs 1001.0 g

[See original for figures.]

Tare  
Net  
Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Dissolution Conditions  
Dissolutest Sotax GAL 091

Temperature: 37°C ± 0.5°C [initials] 7/9/97 [initials] 7/9/97  
Speed: 75 TPM [initials] 7/9/97 [initials] 7/9/97

[initials] 7/9/97  
FOURNIER 1001814

112

5. Readings  
on photospectrometer Gal 233 Chronometer GAL 124  
2 mm tank - 290 nm

LF 178TER LOT RG 2395/01 14 KG LSNA 0.025M 75 TPM

Lambda      No.      Value\_E

[See original for figures and text in English.]

[initials] 7/9/97

FOURNIER 1001815

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF178TER RG 2395/01 at 14 kg LSNa 0.025 M 75 tpm
DATE	07/09/97	NOTEBOOK NO.	LF 178 no. 2, p. 110
APPARATUS	GAL 233 GAL 091	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.025 M
TANK in mm	2	AGITATION	75 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	Volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/9/97  
FOURNIER 1001816

114  
7/17/97

LF178Ter Tablets  
Lot CO197 – 160 mg  
LSNa 0.025 M – 50 TPM  
[Original entry crossed out and initialed.]

1. Preparation of the Dissolution Medium      Balance GAL 065

Water

Weight of Water

[See original for figures.]

[See original for figures.]

Tare  
Net  
Gross

For a 0.025M medium  
[See original for figures.]

[initials] 7/22/97

FOURNIER 1001817



2. Filling of Dissolution Bowls Balance GAL 065  
1 liter of 0.025M LSNa weighs 1001.0 g

[See original for figures.]

Tare  
Net  
Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Dissolution Conditions  
Dissolutest Sotax AT7 GAL 091

Temperature: 37°C ± 0.5°C	[initials] 7/18/97	[initials] 7/18/97
Speed: 50 TPM	[initials] 7/18/97	[initials] 7/18/97

[initials] 7/22/97

FOURNIER 1001818

116

5. Readings  
on photospectrometer Gal 233 Chronometer GAL 124

LF 178TER LOT C0197 0.025M 50TPM

[initials]

Lambda	No.	Value_E
--------	-----	---------

[See original for figures and text in English.]

[initials] 7/22/97

FOURNIER 1001819

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF178TER Tablets Lot
DATE	07/18/97	NOTEBOOK NO.	CO197 LSNa 0.025M 50 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 Ter no. 2 p.114
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.025 M
			50 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	Volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/22/97  
FOURNIER 1001820

118  
7/18/97

LF178Ter Dissolution  
Lot CO197 – 160 mg  
0.025 M – 90 TPM

3. Preparation of the Dissolution Medium  
See page [illegible].

2. Filling of Dissolution Bowls Balance GAL 065  
1 liter of LSNa 0.025 M weighd 1001.0g.  
[See original for figures.]

Tare  
Net  
Gross

3. Weight of Tablets Balance GAL 205  
[See original for figures.]

[initials] 7/22/97  
FOURNIER 1001821

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4. Conditions

Dissolutest GAL 091

Temperature:  $37^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$  [initials] 7/18/97 [initials] 7/18/97

Speed: 90 TPM [initials] 7/18/97 [initials] 7/18/97

LF 178 TER LOT C0197 0.025M 90TPM

Lambda No. Value\_E

[See original for figures.]

5. Readings

on photospectrometer Gal 233

Chronometer GAL 122

FOURNIER 1001822

[initials]

DISSOLUTION[See original for file name.]  
version dated: 2/6/97CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF178TER Tablets Lot CO197 LSNa 0.025M 90 tpm
DATE	07/18/97	NOTEBOOK NO.	LF 178 Ter no. 2 p.118
APPARATUS	GAL 233 GAL 091	FILE	[See original for file name.]
WAVELENGTH	290 nm	ELUANT	LSNa 0.025
TANK in mm	2	AGITATION	90 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l
DISSOLUTION READINGS

volume sampled in ml

TIME POINT	Volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/22/97  
FOURNIER 1001823

7/21/97

LF178Ter Tablets  
Lot CO197 – 160 mg  
0.025 M – 120 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065

Water

Weight of Water

[See original for figures.]

[See original for figures.]

Tare  
Net  
Gross

For a 0.025M medium  
[See original for figures.]

FOURNIER 1001824

Highly Confidential Subject to Protective Order

122

2. Filling of Dissolution Bowls Balance GAL 065  
1 liter of 0.025M LSNa weighs 1001.0 g

[See original for figures.]

Tare  
Net  
Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Conditions  
Dissolutest Sotax AT7 GAL 091

Temperature: 37°C ± 0.5°C	[initials] 7/21/97	[initials] 7/21/97
Speed: 120 TPM	[initials] 7/21/97	[initials] 7/21/97

[initials] 7/22/97  
FOURNIER 1001825



5. Reading  
on photospectrometer Gal 233 Chronometer GAL 122

LF 178TER LOT CO197 LSNA 0.025M 120 TPM

Lambda      No.      Value\_E

[See original for figures and text in English.]

[illegible] Results  
page 127

FOURNIER 1001826

124  
7/21/97

LF178Ter Dissolution  
Lot RG 2395/01 – 14 kg  
LSNa 0.025 M – 50 TPM

1. Preparation of the Dissolution Medium

See page 121.

2. Filling of Dissolution Bowls Balance GAL 065

1 liter of 0.025M LSNa weighs 1001.0 g

[See original for figures.]

Tare  
Net  
Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

FOURNIER 1001827

Highly Confidential Subject to Protective Order

4. Dissolution Conditions  
Dissolutest Sotax AT7 GAL 091

Temperature: 37°C  $\pm$  0.5°C [initials] 7/18/97 [initials] 7/18/97  
Speed: 50 TPM [initials] 7/18/97 [initials] 7/18/97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.025 M 50 TPM

[initials]  
Lambda No. Value\_E

[See original for figures.]

5. Readings  
on photospectrometer Gal 233  
Chronometer GAL 124

FOURNIER 1001828

Speed=50 TPM      [initials] 7/21/97      [initials] 7/21/97

LF 178 TER RG 2395/01 14 KG LSNA 0.025 M 50 TPM

	Lambda	No.	Value_E
5. <u>Reading</u>	[See original for figures.]		
Photospectrometer GAL 233			
Chronometer GAL 122			

[initials] 7/22

FOURNIER 1001829

Highly Confidential Subject to Protective Order

126  
**DISSOLUTION**  
[See original for file name.]  
version dated: 2/6/97

**CONDITIONS AND OPERATING PROCEDURES**

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot RG 2395/01
DATE	07/21/97	NOTEBOOK NO.	14 kg LSNa 0.025M 50 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 124
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.025 M
			50 TPM

**SAMPLE PREPARATION**

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/l

**DISSOLUTION READINGS**

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

\* No significant impact on the results [initials] July 22, 1997

**RESULTS — % DISSOLVED**

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

**RESULTS — QUANTITY DISSOLVED**

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/22/97  
FOURNIER 1001830

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot RG 2395/01
DATE	07/21/97	NOTEBOOK NO.	14 kg LSNa 0.025M 120 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n <sub>2</sub> p 121
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.025 M
			120 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/22/97  
FOURNIER 1001831

128  
7/21/97

LF178Ter Dissolution  
Lot CO197 – 160 mg  
0.05 M – 50 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065

WATER      Weight of the Water

[See original for figures.]

Tare

Net

Gross

Amount of Water Weighed

[See original for figures.]

Amount of LSNa To Be Weighed for 0.05 M Medium

[See original for figures.]

[initials] 7/22/97  
FOURNIER 1001832

Highly Confidential Subject to Protective Order

2. Weight of Dissolution Bowls Balance GAL 065  
1 liter of LSNa 0.05 M weighs 1002.0g  
[See original for figures.]

Tare  
Net  
Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Conditions  
Dissolutest GAL 091

Temp.= 37°C ± 0.5°

Speed= 50 TPM

[initials] 7/22/97

[initials] 7/22/97

[initials] 7/22/97

[initials] 7/22/97

FOURNIER 1001833



130

5. Reading

Photospectrometer GAL 233 – Chronometer GAL 122

LF 178 TER LOT CO197 LSNA 0.05M 50 TPM

Lambda      No.      Value\_E

[See original for figures and text in English.]

[initials] 7/22/97

FOURNIER 1001834

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot RG 2395/01
DATE	07/22/97	NOTEBOOK NO.	14 kg LSNa 0.025M 50 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 128
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.05 M
			50 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/l

[initials]

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001835

Highly Confidential Subject to Protective Order

132  
7/22/97

LF178Ter Dissolution  
Lot CO197 – 160 mg  
0.05 M – 75 TPM

1. Preparation of the Dissolution Medium

See page 128.

2. Weight of Dissolution Bowls      Balance GAL 065

1 liter of LSNa 0.05 M weighs 1002.0g

[See original for figures.]

Tare

Net

Gross

3. Weight of Tablets      Balance GAL 205

[See original for figures.]

FOURNIER 1001836

4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5°

Speed= 75 TPM

[initials] 7/22/97

[initials] 7/22/97

[initials] 7/22/97

[initials] 7/22/97

LF 178 TER LOT CO197 LSNA 0.05M 75 TPM

[initials]

Lambda No. Value\_E

[See original for figures.]

5. Reading

Photospectrometer GAL 233

Chronometer GAL 122

[initials] 7/22/97

FOURNIER 1001837

Highly Confidential Subject to Protective Order

134

DISSOLUTION

[See original for file name.]

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot C0197 LSNa
DATE	07/22/97	NOTEBOOK NO.	0.025M 75 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 132
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.05 M
			75 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion	[See original for figures.]					
Quantity of active ingredient						

Control 100  
mg/lDISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

[initials] 7/22/97

FOURNIER 1001838

Highly Confidential Subject to Protective Order

7/22/97

135

LF178Ter  
Lot CO197 – 160 mg  
0.05 M – 90 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065

WATER

[See original for figures.]

Tare

Net

Gross

Amount of Water Weighed

[See original for figures.]

Amount of LSNa To Be Weighed for 0.05 M Medium

[See original for figures.]

FOURNIER 1001839

Highly Confidential Subject to Protective Order

136

2. Filling of Dissolution Bowls Balance GAL 065

1 liter of LSNa 0.05 M weighs 1002.0g

[See original for figures.]

Tare

Net

Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Conditions

Dissolutest GAL 091

Temp.=  $37^{\circ}\text{C} \pm 0.5^{\circ}$

[initials] 7/23/97

[initials] 7/23/97

Speed= 90 TPM

[initials] 7/23/97

[initials] 7/23/97

[initials] 7/23/97

FOURNIER 1001840

Highly Confidential Subject to Protective Order

5. Reading

Photospectrometer GAL 233 – Chronometer GAL 122

LF 178 TER LOT CO197 LSNA 0.05M 90 TPM

Lambda	No.	Value_E
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[See original for figures.]

FOURNIER 1001841

Highly Confidential Subject to Protective Order



138

DISSOLUTION

{See original for file name.}

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot C0197 LSNa
DATE	07/23/97	NOTEBOOK NO.	0.05M 90 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 135
WAVELENGTH	290 nm	ELUANT	{See original for file name.}
TANK in mm	2	AGITATION	LSNa 0.05 M
			90 TPM

SAMPLE PREPARATION

theoretical mass	{See original for figures.} in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	{See original for figures.}					

Control 100  
mg/lDISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
{See original for figures.}							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
{See original for figures.}									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
{See original for figures.}									

% dissolved

{See original for graph.}

time

quantity dissolved

{See original for graph.}

time

FOURNIER 1001842

Highly Confidential Subject to Protective Order

7/23/97

LF178Ter Dissolution  
Lot RG2395/01 – 14 kg  
0.05 M– 50 TPM

1. Preparation of the Dissolution Medium  
See page 135
2. Filling of the Dissolution Bowls      Balance GAL 065  
1 liter of LSNa 0.05 M weighs 1002.0g  
[See original for figures.]  
    Tare  
    Net  
    Gross
3. Weight of Tablets      Balance GAL 205  
[See original for figures.]

FOURNIER 1001843

140

4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5°

[initials] 7/23/97

[initials] 7/23/97

Speed= 50 TPM

[initials] 7/23/97

[initials] 7/23/97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.05M 50 TPM

Lambda      No.      Value\_E  
[See original for figures.]

Photospectrometer GAL 233

Chronometer GAL 122

[initials] 7/24/97

FOURNIER 1001844

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot RG 2395/01
DATE	07/23/97	NOTEBOOK NO.	14 kg LSNa 0.05M 50 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 139
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.05 M
			50 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion	[See original for figures.]					
quantity of active ingredient						

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001845

Highly Confidential Subject to Protective Order

142  
7/23/97

Lipidil Micro 200  
Lot 97 Canada  
LSNa 0.1 M – 90 TPM

1. Preparation of the Dissolution Medium      Balance GAL 070

WATER

[See original for figures.]

Tare  
Net  
Gross

Amount of Water Weighed  
[See original for figures.]

Amount of LSNa To Be Weighed for 0.1 M Medium  
[See original for figures.]

[initials] 7/24/97

FOURNIER 1001846

2. Filling of Dissolution Bowls Balance GAL 070  
1 liter of LSNa 0.1 M weighs 1004.0g  
[See original for figures.]

Tare  
Net  
Gross

3. Weight of Capsules Balance GAL 205

[See original for figures.]

4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5°

Speed= 90 TPM

[initials] 7/24/97

[initials] 7/24/97

[initials] 7/24/97

[initials] 7/24/97

[initials] 7/24/97

FOURNIER 1001847

144

5. Reading

Photospectrometer GAL 233

Chronometer GAL 122

LIPIDIL MICRO CAPSULES LOT 49 CANADA LSNA 0.1M 90 TPM

Lambda      No.      Value\_E

[See original for figures.]

[illegible initials and date]

FOURNIER 1001848

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	Lipidil Capsules Micro Lot 49
DATE	07/24/97	NOTEBOOK NO.	CanadaLSNa 0.1M 90 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 142
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.1 M
			90 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

[initials]

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001849

Highly Confidential Subject to Protective Order



146  
7/24/97

LF178Ter Dissolution  
Lot CO197 – 160 mg  
0.1 M – 50 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065  
See page 142.

2. Filling of Dissolution Bowls      Balance GAL 065  
1 liter of LSNa 0.1 M weighs 1004.0g  
[See original for figures.]  
Tare  
Net  
Gross

3. Weight of Tablets      Balance GAL 205  
[See original for figures.]

FOURNIER 1001850

4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5°

[initials] 7/24/97

[initials] 7/24/97

Speed= 50 TPM

[initials] 7/24/97

[initials] 7/24/97

5. Reading

Photospectrometer GAL 233

Chronometer GAL 122

LF 178 TER LOT CO197 LSNA 0.1M 50 TPM

Lambda      No.      Value\_E

[See original for figures and text in English.]

FOURNIER 1001851

Highly Confidential Subject to Protective Order

148

[initials]

DISSOLUTION

[See original for file name.]

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot C0197 LSNa
DATE	07/24/97	NOTEBOOK NO.	0.1M 50 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 146
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.1 M
			50 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/lDISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001852

Highly Confidential Subject to Protective Order

7/24/97

LF178Ter Dissolution  
Lot CO197 – 160 mg  
0.1M – 75 TPM

1. Preparation of the Dissolution Medium      Balance GAL [illegible]

Weight of the Water

WATER

[See original for figures.]

Tare

Net

Gross

Amount of Water Weighed

[See original for figures.]

Amount of LSNa To Be Weighed for 0.1 M Medium

[See original for figures.]

FOURNIER 1001853

Highly Confidential Subject to Protective Order

150

2. Filling of Dissolution Bowls Balance GAL 065

1 liter of LSNa 0.1 M weighs 1004.0g

[See original for figures.]

Tare

Net

Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5°

[initials] 7/25/97

[initials] 7/25/97

Speed= 75 TPM

[initials] 7/25/97

[initials] 7/25/97

FOURNIER 1001854

Highly Confidential Subject to Protective Order

5. Reading

Photospectrometer GAL 233

Chronometer GAL 122

LF 178 TER LOT CO197 LSNA 0.1M 75 TPM

Lambda      No.      Value\_E

[See original for figures.]

FOURNIER 1001855

Highly Confidential Subject to Protective Order

152

[initials]

DISSOLUTION

[See original for file name.]

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot C0197 LSNa
DATE	07/25/97	NOTEBOOK NO.	0.1M 75 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 149
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.1 M
			75 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/lDISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001856

Highly Confidential Subject to Protective Order

7/28/97

LF178Ter Dissolution  
Lot CO197 – 160 mg  
0.1 – 120 TPM

Pagination error. See 1<sup>st</sup> page.  
[initials]

[Original entry crossed out and initialed.]

1. Preparation of the Dissolution Medium Balance GAL 065

WATER Weight of the Water

[See original for figures.]

Tare

Net

Gross

Amount of Water Weighed

[See original for figures.]

Amount of LSNa for 0.1 M Medium

[See original for figures.]

FOURNIER 1001857

Highly Confidential Subject to Protective Order



153

2. Filling of Dissolution Bowls Balance GAL 065

1 liter of LSNa 0.1 M weighs 1004.0g

[See original for figures.]

Tare

Net

Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5°

[initials] 7/28/97

[initials] 7/28/97

Speed= 120 TPM

[initials] 7/28/97

[initials] 7/28/97

FOURNIER 1001858

Highly Confidential Subject to Protective Order

5. Reading

Photospectrometer GAL 233

Chronometer GAL 122

LF 178 TER LOT CO197 LSNA 0.1M 120 TPM

[initials]

Lambda      No.      Value\_E

[See original for figures.]

FOURNIER 1001859

Highly Confidential Subject to Protective Order

155

DISSOLUTION

[See original for file name.]

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot C0197 LSNa
DATE	07/28/97	NOTEBOOK NO.	0.1M 120 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 152
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.1 M
			120 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/lDISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001860

Highly Confidential Subject to Protective Order

7/28/97

LF178Ter Dissolution  
Lot RG 2395/01 – 14 kg  
0.1M – 50 TPM

1. Preparation of the Dissolution Medium  
See page 152.

2. Weight of Dissolution Bowls      Balance GAL 065  
[See original for figures.]  
Tare  
Net  
Gross  
1 liter of LSNa 0.1M weighs 1004.0 g.

3. Weight of Tablets      Balance GAL 205  
[See original for figures.]

FOURNIER 1001861

157

4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5

[initials] 7/28/97

[initials] 7/28/97

Speed= 50 TPM

[initials] 7/28/97

[initials] 7/28/97

LF TER LOT RG 2395/01 14 KG LSNA 0.1M 50 TPM

Lambda      No.      Value\_E

[See original for figures.]

FOURNIER 1001862

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot RG 2395/01
DATE	07/28/97	NOTEBOOK NO.	14 kg LSNa 0.1M 50 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 156
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.1 M
			50 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/lDISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001863

Highly Confidential Subject to Protective Order

159

7/28/97

LF178Ter Dissolution  
RG 2395/01 – 14 kg  
LSNa 0.05 M – 75 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065

WATER      Weight of the Water

[See original for figures.]

Tare

Net

Gross

Amount of Water Weighed

[See original for figures.]

Amount of LSNa To Be Weighed for 0.05 M Medium

[See original for figures.]

FOURNIER 1001864

Highly Confidential Subject to Protective Order

2. Filling of Dissolution Bowls Balance GAL 065  
1 liter of LSNa 0.05 M weighs 1002.0g  
[See original for figures.]

Tare  
Net  
Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Conditions

Dissolutest GAL 091

Temp.=  $37^{\circ}\text{C} \pm 0.5^{\circ}$

[initials] 7/28/97

[initials] 7/28/97

Speed= 75 TPM

[initials] 7/28/97

[initials] 7/28/97

FOURNIER 1001865

Highly Confidential Subject to Protective Order



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5. Reading

Photospectrometer GAL 233

Chronometer GAL 122

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.05M 75 TPM

[initials]

Lambda      No.      Value\_E

[See original for figures.]

FOURNIER 1001866

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot RG 2395/01
DATE	07/29/97	NOTEBOOK NO.	14 kg LSNa 0.05M 75 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 159
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.05 M
			75 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

[initials]

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001867

Highly Confidential Subject to Protective Order

163  
7/29/97

LF178Ter Dissolution  
Lot RG 2395/01 – 14 kg  
LSNa 0.05 M – 90 TPM

1. Preparation of the Dissolution Medium  
See page 159.
2. Filling of Dissolution Bowls      Balance GAL 065  
1 liter of LSNa 0.05 M weighs 1002.0g  
[See original for figures.]  
    Tare  
    Net  
    Gross

3. Weight of Tablets      Balance GAL 205  
  
[See original for figures.]

FOURNIER 1001868

4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5°

[initials] 7/29/97

[initials] 7/29/97

Speed= 90 TPM

[initials] 7/29/97

[initials] 7/29/97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.05M 90 TPM

Lambda      No.      Value\_E

[See original for figures.]

5. Reading

Photospectrometer GAL 233

Chronometer GAL 122

FOURNIER 1001869

165

DISSOLUTION

[See original for file name.]

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot RG 2395/01
DATE	07/29/97	NOTEBOOK NO.	14 kg LSNa 0.05M 90 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 163
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.05 M
			90 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/lDISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001870

Highly Confidential Subject to Protective Order

7/29/97

LF178Ter Dissolution  
Lot TG 192 – 18 kg  
LSNa 0.025 M – 75 TPM

1. Preparation of the Dissolution Medium      Balance GAL 065

WATER      Weight of the Water

[See original for figures.]

Tare

Net

Gross

Amount of Water Weighed

[See original for figures.]

Amount of LSNa To Be Weighed for 0.025 M Medium

[See original for figures.]

FOURNIER 1001871

167

2. Filling of Dissolution Bowls Balance GAL 065

1 liter of LSNa 0.025 M weighs 1001.0g

[See original for figures.]

Tare

Net

Gross

3. Weight of Tablets Balance GAL 205

[See original for figures.]

4. Conditions

Dissolutest GAL 091

Temp.=  $37^{\circ}\text{C} \pm 0.5^{\circ}$

[initials] 7/30/97

[initials] 7/30/97

Speed= 75 TPM

[initials] 7/30/97

[initials] 7/30/97

FOURNIER 1001872

Highly Confidential Subject to Protective Order

5. Reading

Photospectrometer GAL 233

Chronometer GAL 122

LF 178 TER LOT TG 192 18 KG LSNA 0.025M 75 TPM

[initials]

Lambda	No.	Value_E
[See original for figures.]		

FOURNIER 1001873

Highly Confidential Subject to Protective Order



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DISSOLUTION

[See original for file name.]

version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot TG 192 18 kg
DATE	07/30/97	NOTEBOOK NO.	LSNa 0.025M 75 tpm
APPARATUS	GAL 233 GAL 091	FILE	LF 178 TER n2 p 166
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.025 M
			75 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

mass of test portion  
quantity of active ingredient

CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]					

Control 100  
mg/l

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001874

Highly Confidential Subject to Protective Order

7/30/97

LF178Ter Dissolution  
Lot RG 2395/01 – 14 kg  
LSNa 0.025 – 120 TPM

1. Preparation of the Dissolution Medium  
See page 166.
2. Filling of Dissolution Bowls      Balance GAL 065  
1 liter of LSNa 0.025 M weighs 1001.0g  
[See original for figures.]  
    Tare  
    Net  
    Gross

3. Weight of Tablets      Balance GAL 205  
[See original for figures.]

FOURNIER 1001875

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4. Conditions

Dissolutest GAL 091

Temp.= 37°C ± 0.5°                      [initials] 7/30/97                      [initials] 7/30/97

Speed= 120 TPM                              [initials] 7/30/97                      [initials] 7/30/97

LF 178 TER LOT RG 2395/01 14 KG LSNA 0.025M 120 TPM

[initials]

Lambda              No.      Value\_E

[See original for figures.]

Reading

Photospectrometer GAL 233

Chronometer GAL 122

FOURNIER 1001876

Highly Confidential Subject to Protective Order

[FOURNIER 1001877 is a duplicate of FOURNIER 1001876 and shows the table in greater detail.]

FOURNIER 1001877

Highly Confidential Subject to Protective Order

DISSOLUTION

[See original for file name.]  
version dated: 2/6/97

CONDITIONS AND OPERATING PROCEDURES

OPERATOR	D. LECRIT	TITLE	LF 178 TER lot RG 2395/01
DATE	07/30/97	NOTEBOOK NO.	14 kg LSNa 0.025M 120 tpm
APPARATUS	GAL 233, GAL 091	FILE	LF 178 TER n2 p 170
WAVELENGTH	290 nm	ELUANT	[See original for file name.]
TANK in mm	2	AGITATION	LSNa 0.025 M
			75 TPM

SAMPLE PREPARATION

theoretical mass	[See original for figures.] in mg
theoretical dose	

	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
mass of test portion quantity of active ingredient	[See original for figures.]					

Control 100  
mg/l

[initials]

DISSOLUTION READINGS

volume sampled in ml

TIME POINT	volume in ml	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6
[See original for figures.]							

RESULTS — % DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

RESULTS — QUANTITY DISSOLVED

TIME POINT	MEAN	CELL 1	CELL 2	CELL 3	CELL 4	CELL 5	CELL 6	standard deviation	CV
[See original for figures.]									

% dissolved

[See original for graph.]

time

quantity dissolved

[See original for graph.]

time

FOURNIER 1001878

Highly Confidential Subject to Protective Order

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[This page is crossed out.]  
[initials] 7/30/97

FOURNIER 1001879

Highly Confidential Subject to Protective Order

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[initials] 7/30/97

FOURNIER 1001880

Highly Confidential Subject to Protective Order

[This page is crossed out.]  
[initials] 7/30/97

FOURNIER 1001881

Highly Confidential Subject to Protective Order



[This page is crossed out.]  
[initials] 7/30/97

FOURNIER 1001882

Highly Confidential Subject to Protective Order

[This page is crossed out.]  
[initials] 7/30/97

FOURNIER 1001883

Highly Confidential Subject to Protective Order

Page	Date	Title
[See original for figures.]	07/03/97	[See original for figures, except for line 12, which reads as follows:]
	07/08/97	
	07/09/97	
	07/18/97	
	07/18/97	
	07/21/97	
	07/21/97	
	07/21/97	
	07/22/97	
	07/22/97	
	07/23/97	
	07/23/97	
	07/24/97	Dissolution Lipidil Capsule Lot 49 Canada LSNa 0.1 – 90 TPM
	07/24/97	
	07/28/97	
	07/28/97	
	07/28/97	
	07/29/97	
	07/29/97	
	07/30/97	

FOURNIER 1001884

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Page	Date	Subject
[See original for figures.]	05/15/97	Dissolution of Lipidil 200 Capsules- Lot 37 Canada 0.025M/ 75
	05/16/97	Lipidil 200 Capsules – Lot 39 Canada 0.025M/75
	05/16/97	Lipidil 200 Capsules – Lot 40 Canada 0.025 M/75 TPM
	06/03/97	[See original for figures.]
	06/03/97	
	06/05/97	
	06/09/97	
	06/10/97	
	06/11/97	
	06/12/97	
	06/12/97	
	06/13/97	Idem p. 41 – [illegible] with and without filtration
	06/17/97	[See original for figures.]
	06/17/97	
	06/18/97	
	06/18/97	
	06/19/97	
	06/19/97	
	06/20/97	
	06/24/97	
	06/24/97	Dissolution Lot 158 tablets 0.025 75 TPM
	06/25/97	
	06/27/97	
	06/30/97	
	07/01/97	
	07/01/97	
	07/03/97	

FOURNIER 1001885

[illegible] Patrick Nourissat, notary and member of [illegible], Patrick Nourissat, Didier Nourissat and Hugues Misserey, holder of a notary post headquartered at Duon, 25 rue Buffon.

IN WITNESS WHEREOF having attending the adjournment of this document comprising 179 pages, duly reviewed and paraphrased on the last page and to be stored in the a[initials]hives of formulations of medicines for Laboratoires Fournier, at Centre de Biogalenique, 42 rue de Longvic in Chenôve.

WITH THE SEAL OF OFFICE DULY AFFIXED  
IN DUON  
ON JUNE 10, 2002

[seal]

FOURNIER 1001886

Highly Confidential Subject to Protective Order

# **Exhibit S**

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

## ANALYSIS CERTIFICATE

PRODUCT. . . . . : LIPANTIL MICRO.M.200 MG.28 GEL.MV.GB  
BATCH No . . . . . : 71686  
DATE OF MANUFACTURING . . . . . : 7/03/02  
EXPIRY DATE. . . . . : July 2005  
SPECIFICATION No. . . . . : 06-50250/3

TESTS	SPECIFICATIONS	RESULTS
Characteristics	Opaque, orange capsule, size 1 containing a whitish powder	Complies
Average mass	350.0 mg +/- 5 %	357.6 mg
Mass uniformity	Complies with the requirements of European Pharmacopoeia (4:7.5)	Complies
Disintegration time	<or= 15 min	9.5 min
Dissolution after 20 min	75.0 to 95.0 %	97.2 %
Dissolution after 40 min	>or= 90.0 %	102.1 %
Identification of fenofibrate	Retention time of the main peak identical with that of the reference	Complies
Assay of fenofibrate	190. to 210.0 mg per capsule	200.5 mg
Impurity A	<or= 0.05 %	} not performed on a routine basis
Impurity B	<or= 0.05 %	
Impurity C	<or= 0.05 %	
Impurity D	<or= 0.05 %	
Impurity E	<or= 0.10 %	
Impurity F	<or= 0.05 %	
Impurity G	<or= 0.20 %	
Unknown impurities	<or= 0.05 % each	
Total of impurities	<or= 0.40 %	
Microbiologie (Ph. Eur.)	Periodic control	Non/No
Bacteria	<or= 1000 /g	
Fungi	<or= 100 /g	
Escherichia coli	Absence in 1g	

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

Micheline ROUSSEAU

DATE

8/05/02

Highly Confidential –  
Subject to Protective Order

FOURNIER 0029743

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: STEARATE DE MG ORIGINE VEGETALE PE
SUPPLIER . . . . .	: QUIMDIS
SUPPLIER BATCH . . . . .	: 205848
PRODUCT CODE . . . . .	: 601201
EXPIRY DATE . . . . .	: 6/12/05
SPECIFICATION . . . . .	: 01-00004/9

=====


INTERNAL BATCH N° : 65747	DATE OF RECEIPT : 6/12/02
---------------------------	---------------------------

TESTS	SPECIFICATIONS	RESULTS
Characters	A white, very fine, light powder, greasy to the touch	Complies
Solubility	Practically insoluble in water and in ethanol	Complies
Identification C: GC	Retention times of stearic and palmitic acids identical to the reference solution	Complies
Identification D : magnesium	Positive reaction of magnesium	Complies
Acidity ou alcalinity	<or= 0.5ml HCl (0.01M) or NaOH (0.01M)	Complies
Chlorides	<or= 0.1 %	Complies
Sulphates	<or= 0.5 %	Complies
Cadmium	<or= 3 ppm	Complies
Lead	<or= 10 ppm	Complies
Nickel	<or= 5 ppm	Complies
Loss on drying	<or= 6.0 %	4.8 %
Assay of magnesium (calculated on the dried basis)	4.0 to 5.0 %	4.9 %
Stearic acid	>or= 40.0 %	66.5 %
Sum of stearic acide and palmitic acid	>or= 90.0 %	99.3 %
Total viable aerobic count	<or= 1 000 /g	4 /g
Escherichia coli	Absence in 1 g	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

-----  
  
Micheline ROUSSEAU

-----  
6/27/02

**Highly Confidential –  
Subject to Protective Order**

**FOURNIER 0029920**



# **Exhibit P**

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des Prés Potets  
21121 Fontaine les Dijon  
FRANCE

## ANALYSIS CERTIFICATE

=====

PRODUCT. . . . .	LIPANTIL MICRO.M.67 MG.90 GEL.MV.GB
BATCH N° . . . . .	68128
DATE OF MANUFACTURING. . . . .	9/11/01
EXPIRY DATE. . . . .	September 2004
SPECIFICATION N° . . . . .	06-50251/1

=====

TESTS	SPECIFICATIONS	RESULTS
Characteristics	Opaque, yellow capsule size 4 containing a whitish powder	Complies
Content average mass	111.5 to 123.0 mg	117.5 mg
Mass uniformity	Complies with the requirements of the EP (+/- 10%)	Complies
Disintegration time	<or= 15 min	4 min
Dissolution after 20 min	75.0 to 95.0 %	94.0 %
Dissolution after 40 min	>or= 90.0%	99.9 %
Identification of fenofibrate	Retention time of the main peak identical with that of the reference	Complies
Assay of impurities	63.6 to 70.4 mg	67.2 mg
Impurity A	<or= 0.05 %	0.00 %
Impurity B	<or= 0.05 %	0.00 %
Impurity C	<or= 0.05 %	0.00 %
Impurity D	<or= 0.05 %	0.00 %
Impurity E	<or= 0.10 %	0.01 %
Impurity F	<or= 0.05 %	0.00 %
Impurity G	<or= 0.20 %	0.14 %
Unknown impurities	<or= 0.05% each	0.00 %
Total impurities	<or= 0.40 %	0.15 %
Microbiologie (Ph. Eur.)	Periodic control	Non/No
Bacteria	<or= 1000 /g	
Fungi	<or= 100 /g	
Escherichia coli	Absence in 1g	

This batch has been released by the Qualified Person in accordance with the current European GMP-requirements

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

Date

12/18/01

Micheline ROUSSEAU

Highly Confidential –  
Subject to Protective Order

FOURNIER 0029742

Laboratoires Fournier S.A. - Rue des Prés Potets - 21121 Fontaine-les-Dijon  
Tél. 03 80 44 78 00 - Fax 03 80 44 78 18  
Laboratoires Fournier S.A. - Siège Social - 42, rue de Longvic - 21300 Chenôve - France  
S.A. au Capital de 18 035 296 euros - RCS Dijon B 311 596 670 - TVA FR 52 311 596 670

LABORATOIRES  
FOURNIER

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT	: MAGNESIUM STEARATE
SUPPLIER	: FACI / QUARRECHIM
SUPPLIER BATCH	: MGS10147
PRODUCT CODE	: 402595
INTERNAL BATCH N°	: 63581
EXPIRY DATE	: 7/09/04
DATE OF RECEIPT	: 7/09/01
SPECIFICATION	: 01-00004/9

=====

TESTS	SPECIFICATIONS	RESULTS
Characters	A white, very fine, light powder, greasy to the touch	Complies
Solubility	Practically insoluble in water and in ethanol	Complies
Identification C: GC	Retention times of stearic and palmitic acids identical to the reference solution	Complies
Identification D : magnesium	Positive reaction of magnesium	Complies
Acidity ou alcalinity	<or= 0.5ml HCl (0.01M) or NaOH (0.01M)	Complies
Chlorides	<or= 0.1 %	Complies
Suiphates	<or= 0.5 %	Complies
Cadmium	<or= 3 ppm	Complies
Lead	<or= 10 ppm	Complies
Nickel	<or= 5 ppm	Complies
Loss on drying	<or= 6.0 %	2.6 %
Assay of magnesium (calculated on the dried basis)	4.0 to 5.0 %	4.7 %
Stearic acid	>or= 40.0 %	65.4 %
Sum of stearic acide and palmitic acid	>or= 90.0 %	94.0 %
Total viable aerobic count	<or= 1 000 /g	4 /g
Escherichia coli	Absence in 1 g	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

=====

Micheline ROUSSEAU

=====  
8/06/01

**FOURNIER 0029922**

**Highly Confidential -  
Subject to Protective Order**

# **Exhibit H**



LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

ANALYSIS CERTIFICATE

=====

PRODUCT. . . . .	:	LIPIDIL.M.67 MG.1000 GEL.VRC.CA
BATCH No . . . . .	:	76
DATE OF MANUFACTURING . . . . .	:	9/29/97
EXPIRY DATE. . . . .	:	September 2000
SPECIFICATION No. . . . .	:	06-50240/1

=====

TESTS	SPECIFICATIONS	RESULTS
=====	=====	=====
Capsules characteristics	Opaque ,yellow capsule ,size 4 containing a whitish powder	Complies
Identification of fenofibrate (HPLC)	Retention time of the main peak identical with that of the reference	Complies
Average mass	111.5 to 123.0 mg	118.6 mg
Disintegration time	<or= 15 min	5.5 min
Mass uniformity	Complies with USP requirements	Complies
Dissolution	Q40 minutes = 90 %	100.66 %
Total bacterial count	<or= 1 000 /g	20 /g
Escherichia coli	Absence in 10 g	Complies
Assay of fenofibrate	63.6 to 70.4 mg per capsule	66.1 mg/cap

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

10/27/97

Micheline ROUSSEAU

FOURNIER 1005219

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Subject to  
Protective Order



Laboratoires Fournier S.A. - Rue des Prés Potets - 21121 Fontaine-les-Dijon  
Tél. 03 80 44 78 00 - Fax 03 80 44 78 38  
Laboratoires Fournier S.A. - Siège Social - 42 rue de Longvic - 21300 Chenôve - France  
S.A. au Capital de 18 035 296 euros - RCS Dijon B 311 596 670 - TVA FR 52 311 596 670

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: MAGNESIUM STEARATE
SUPPLIER . . . . .	: QUARRE MARCEL ET CIE
SUPPLIER BATCH . . . . .	: MGS60519
PRODUCT CODE . . . . .	: 402597
INTERNAL BATCH N° :	46673
EXPIRY DATE . . . . .	: 11/16/98
DATE OF RECEIPT :	5/16/97
SPECIFICATION . . . . .	: 01-00161/4

=====

TESTS	SPECIFICATIONS	RESULTS
Characteristics	White ,fine ,bulky powder	Complies
Solubility	Insoluble in water, in alcohol and ether	Complies
Identification A	Responds to the test for magnesium	Complies
Identification B : GC	Retention times of stearic and palmitic acids identical to the reference solution	Complies
Acidity or alkalinity	<or= 0.05 ml 0.1N HCl or 0.1N NaOH	Complies
Chloride	<or= 0.1%	Complies
Sulfate	<or= 1.0%	Complies
Lead	<or= 0.001%	Complies
Loss on drying	<or= 6.0 %	3.0 %
Total bacterial count	<or= 1 000/g	4 /g
Yeasts and molds	<or= 500/g	4 /g
Salmonella species	Absence in 10g	Complies
Escherichia coli	Absence in 10g	Complies
Relative content of stearic acid	>or= 40 %	69 %
Relative content of stearic acid and palmitic acid	>or= 90 %	95 %
OVI : Methylene chloride	<or= 500 ppm	} not required
OVI : Chloroform	<or= 50 ppm	
OVI : Benzene	<or= 100 ppm	
OVI : Trichloroethylene	<or= 100 ppm	
OVI : 1,4 Dioxane	<or= 100 ppm	
Assay (expressed in magnesium)	4.0 to 5.0 % (calculated on the dried basis)	4.6 %

CONCLUSION : Complies

FOURNIER 1005220


QUALITY ASSURANCE MANAGER


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6/05/97

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 Fournier

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RAW MATERIAL

ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: LACTOSE EFC	
SUPPLIER . . . . .	: SPCI	
SUPPLIER BATCH . . . . .	: 032/717	
PRODUCT CODE . . . . .	: 402552	INTERNAL BATCH N° : 47057
EXPIRY DATE . . . . .	: 12/10/98	DATE OF RECEIPT : 6/10/97
SPECIFICATION . . . . .	: 01-00011/8	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristic	White to off-white crystalline powder, odourless	Complies
Solubility	Freely through slowly soluble in water, practically insoluble in alcohol	Complies
Identification A : IR spectrum	Conform to standard	Complies
Appearance of solution	Clear, odourless	Complies
Acidity or alcalinity	Colourless with phenolphthaleine ( maximum 0.4 ml NaOH 0.1N)	Complies
Specific rotatory power	54.4 to 55.9 °	55.3 °
Absorbance at 400 nm	<or= 0.04	Complies
Absorbance from 210 to 220 nm	<or= 0.25	Complies
Absorbance from 270 to 300 nm	<or= 0.07	Complies
Heavy metals	<or= 5 ppm	Complies
Water content	4.5 to 5.5 %	5.0 %
Sulphated ash	<or= 0.1 %	0.0 %
Loss on drying	<or= 0.5 %	0.1 %
Total viable microorganisms	<or= 100 /g	4 /g
Escherichia coli	Absence in 1g	Complies
Organic volatil control (USP XXIII)	Periodic control	Oui/Yes
Organic volatil impurity : benzen	<or= 100 ppm	Complies
Organic volatil impurity : chloroform	<or= 50 ppm	Complies
Organic volatil impurity : 1,4 dioxan	<or= 100 ppm	Complies
Organic volatil impurity : methylen chloride	<or= 500 ppm	Complies
Organic volatil impurity : trichloroethy len	<or= 100 ppm	Complies

CONCLUSION : Complies

**FOURNIER 1005221**

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9/03/97



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RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: PREGELATINIZED STARCH	
SUPPLIER . . . . .	: GAZECHIM	
SUPPLIER BATCH . . . . .	: E9076	
PRODUCT CODE . . . . .	: 402103	INTERNAL BATCH N° : 47875
EXPIRY DATE . . . . .	: 1/22/99	DATE OF RECEIPT : 7/22/97
SPECIFICATION . . . . .	: 01-00160/6	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Description	Moderately coarse to fine, white to offwhite powder	Complies
Solubility	Slightly soluble to soluble in cold water ; insoluble in alcohol	Complies
Identification by iodine	Reddish violet to deep blue color	Complies
Salmonellas species	Absence in 10g	Complies
Escherichia coli	Absence in 10g	Complies
pH	4.5 to 7.0	6.1
Loss on drying	<or= 14.0 %	6.6 %
Residue on ignition	<or= 0.5 %	0.3 %
Iron	<or= 0.002%	Complies
Oxidizing substances	No distinct blue, brown or purple color	Complies
Sulfur dioxide	<or= 0.008%	Complies

CONCLUSION : Complies

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8/27/97

  
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RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: YELLOW CAPSULES, SIZE N°4
SUPPLIER . . . . .	: SHIONOGI QUALICAPS
SUPPLIER BATCH . . . . .	: E9701801
PRODUCT CODE. . . . .	: 406774
INTERNAL BATCH N° :	47921
EXPIRY DATE . . . . .	: 7/24/99
DATE OF RECEIPT :	7/24/97
SPECIFICATION. . . . .	: 01-00080/4

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristics	Capsule, size 4, yellow, identical to the reference, smooth envelope, shiny, clean, neither brittle nor fragile	Complies
Gelatin A :	Characteristic gel	Complies
Gelatin B :	Cloudy with picric acid	Complies
Quinoline yellow	Rf identical to the reference	Complies
Erythrosin	Rf identical to the reference	Complies
Sulphur dioxide	<or= 0.1 %	0.1 %
Titanium dioxide	Yellow to orange-yellow coloring in the presence of hydrogen peroxide	Complies
Ferrous oxyde	No blue colouring	Complies
Lubricants	<or= 0.5 %	0.0 %
Loss and drying	12.0 to 16.0 %	14.6 %
Sulphated ash	<or= 9.0 %	2.7 %
Disintegration time	<or= 15 min	6 min

CONCLUSION : Complies

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RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT	: CROSPVIDONE XL10 USP		
SUPPLIER	: ISP		
SUPPLIER BATCH	: N60409		
PRODUCT CODE	: 402699	INTERNAL BATCH N°	: 41192
EXPIRY DATE	: 12/19/96	DATE OF RECEIPT	: 6/19/96
SPECIFICATION	: 01-00180/1		

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characters	Very fine white, tasteless powder	Complies
IR spectrum	Maxima identical to those of control spectrum	Complies
Starch coloration	No blue coloration	Complies
pH of a 1% aqueous suspension	5.0 to 8.0	6.3
Water content	<or= 5.0 %	4.1 %
Sulphated ash	<or= 0.4 %	0.0 %
Water-soluble substances	<or= 1.5 %	0.5 %
Heavy metals	<or= 10 ppm	Complies
Vinylpyrrolidone	<or= 0.1 %	0.0 %
Nitrogen	11.0 to 12.8 %	11.9 %
(relative to dry product)		

CONCLUSION : Complies

-----

QUALITY ASSURANCE MANAGER

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10/04/96

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RAW MATERIAL

ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: COMICRONISAT FENOPIBRATE	
SUPPLIER . . . . .	: PLASTO	
SUPPLIER BATCH . . . . .	: 97667	
PRODUCT CODE . . . . .	: 402701	INTERNAL BATCH N° : 48174
EXPIRY DATE . . . . .	: 8/04/99	DATE OF RECEIPT : 8/29/97
SPECIFICATION. . . . .	: 03-08001/9	

=====

TESTS	SPECIFICATIONS	RESULTS
Caracteres	Poudre tres fine ,blanche et inodore	Complies
Identification du fénofibrate	Rf identique au temoin	Complies
Dosage du laurylsulfate de sodium	3.00 to 3.70 %	
Granulométrie laser :Médiane	5.0 to 15.0 um	
Granulométrie laser :Diamètre des particules	Au moins 90% des particules de diamètre <ou= 35um	

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

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RAW MATERIAL

ANALYSIS CERTIFICATE

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=====
PRODUCT . . . . . : COMICRONISAT FENOPIBRATE
SUPPLIER . . . . . : PLASTO
SUPPLIER BATCH . . . . . : 97690
PRODUCT CODE . . . . . : 402701      INTERNAL BATCH N° : 48329
EXPIRY DATE . . . . . : 9/08/99      DATE OF RECEIPT : 9/09/97
SPECIFICATION. . . . . : 03-08001/9
=====
```

TESTS	SPECIFICATIONS	RESULTS
Caracteres	Poudre tres fine ,blanche et inodore	Complies
Identification du fénofibrate	Rf identique au temoin	Complies
Dosage du laurylsulfate de sodium	3.00 to 3.70 %	
Granulométrie laser :Médiane	5.0 to 15.0 um	
Granulométrie laser :Diamètre des particules	Au moins 90% des particules de diamètre <ou= 35um	


CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

9/23/97

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## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: PURIFIED WATER	
SUPPLIER . . . . .	: PRODUCTION FONTAINE 2	
SUPPLIER BATCH . . . . .	: SALLE 1611	
PRODUCT CODE. . . . .	: 404900	INTERNAL BATCH N° : 1974011
EXPIRY DATE . . . . .	:	DATE OF RECEIPT : 10/03/97
SPECIFICATION. . . . .	: 01-00008/11	

=====

TESTS	SPECIFICATIONS	RESULTS
Characters	Clear liquid, colourless , tasteless	Complies
pH	5.0 to 7.0	6.5
Oxidisable substances	Slightly pinkish with potassium permanganate	Complies
Chlorides	No precipitate for at least 15 min.	Complies
Nitrates	<or= 0.2 ppm	Complies
Sulfates	No precipitate for at least 1 hour	Complies
Ammonium	<or= 0.2 ppm	Complies
Calcium and magnesium	Pure blue colour with mordant black 11R	Complies
Heavy metals	<or= 0.1 ppm	Complies
Residue on evaporation	<or= 0.001 %	0.001 %
Total viable aerobic count	<or= 100 /ml	1 /ml

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

10/10/97

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# **Exhibit E**

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ANALYSIS CERTIFICATE

=====

PRODUCT. . . . .	: LIPIDIL.M.200 MG.1000 GEL.VRC.CA
BATCH No . . . . .	: 52
DATE OF MANUFACTURING . . . . .	: 4/03/97
EXPIRY DATE. . . . .	: April 2000
SPECIFICATION No. . . . .	: 06-50067/5

=====

TESTS	SPECIFICATIONS	RESULTS
=====	=====	=====
Characteristics	Opaque ,orange capsule size 1, printed "Lipidil Micro" containing a whitish powder	Complies
Average mass	350,0 mg +/- 5%	354.3 mg
Uniformity of mass	Complies with the requirements of European Pharmacopoeia (% 7.5)	Complies
Disintegration	<or= 15 min	9 min
Dissolution : 40 min	Q = 90,0 %	96.0 %
Identification of fénofibrate	Retention time of the main peak identical to that of the référence	Complies
Assay of fenofibrate	190,0 à 210,0 mg per capsule	202.5 mg
Bacteria	<or= 1 000 /g	20 /g
Escherichia coli	Absence in 10 g	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

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4/23/97



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RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: CAPSULES, SIZE 1, ORANGE/ORANGE	
SUPPLIER . . . . .	: CAPSUGEL	
SUPPLIER BATCH . . . . .	: C63564	
PRODUCT CODE . . . . .	: 406342	INTERNAL BATCH N° : 44498
EXPIRY DATE . . . . .	: 1/07/99	DATE OF RECEIPT : 1/07/97
SPECIFICATION . . . . .	: 01-00178/3	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristics	Capsule, size n°1, orange/orange identical to the reference, smooth, shiny, clean, with neither brittle nor fragile envelope	Complies
Printing of the capsules	The capsules are printed "LIPIDIL" on one part, MICRO on the other part with black ink	Complies
Identification A	Characteristic gel	Complies
Identification B	Cloudy with picric acid	Complies
Coloring agents : erythrosin	Rf identical to the reference	Complies
Sulphur dioxide	<or= 0.1 %	0.0 %
Titanium dioxide	Yellow to yellow-orange colouring in the presence of hydrogen peroxide	Complies
Ferrous oxides	Blue coloring in the presence of potassium ferrocyanide	Complies
Lubricants	<or= 0.5 %	0.1 %
Loss on drying	12.0 to 16.0 %	13.9 %
Sulphated ash	<or= 9.0 %	3.6 %
Disintegration time	<or= 15 min	3.5 min
Total bacterial count	Not more than 1000/g	4 /g
Total bacterial count	Not more than 1000/g	4 /g
Escherichia coli	Absence in 10g	Complies
Escherichia coli	Absence in 10g	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

1/20/97

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FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

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=====
PRODUCT . . . . . : PREGELATINIZED STARCH
SUPPLIER . . . . . : GAZECHIM
SUPPLIER BATCH . . . . . : E8739
PRODUCT CODE . . . . . : 402103          INTERNAL BATCH N° : 43517
EXPIRY DATE . . . . . : 4/01/98          DATE OF RECEIPT : 10/31/96
SPECIFICATION. . . . . : 01-00160/3
=====
  
```

TESTS	SPECIFICATIONS	RESULTS
Description	Moderately coarse to fine, white to offwhite powder	Complies
Solubility	Slightly soluble to soluble in cold water ; insoluble in alcohol	Complies
Identification by iodine	Reddish violet to deep blue color	Complies
Salmonellas species	Absence in 10g	Complies
Escherichia coli	Absence in 10g	Complies
pH	4.5 to 7.0	5.7
Loss on drying	<or= 14.0 %	6.8 %
Residue on ignition	<or= 0.5 %	0.2 %
Iron	<or= 0.002%	Complies
Oxidizing substances	No distinct blue, brown or purple color	Complies
Sulfur dioxide	<or= 0.008%	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

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11/25/96

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RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: LACTOSE	
SUPPLIER . . . . .	: SPCI	
SUPPLIER BATCH . . . . .	: 032/603	
PRODUCT CODE . . . . .	: 402549	INTERNAL BATCH N° : 43902
EXPIRY DATE . . . . .	: 5/25/98	DATE OF RECEIPT : 11/25/96
SPECIFICATION . . . . .	: 01-00166/3	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Description	White, free-flowing powder	Complies
Solubility	Freely but slowly soluble in water ; pratically insoluble in alcohol	Complies
Identification A : Infrared absorption	Identical with the reference standard	Complies
Identification B : Thin-layer chromatography	Test solution corresponds in appearance and Rf value to that obtained from standard solution	Complies
Identification C : With ammonium hydroxyde at 80°C	Red color	Complies
Clarity and color of solution	Aborbance at 400 nm <or= 0.04	Complies
Acidity or alkalinity	Solution colorless <or= 0.4ml NaOH 0.1N to produce a red color	Complies
Specific rotation / anhydrous basis	54.4 to 55.9 °	54.9 °
Protein and light-absorbing impurities: absorbance from 210 to 220 nm	<or= 0.25	Complies
Absorbance from 270 to 300 nm	<or= 0.07	Complies
Loss on drying	<or= 0.5 %	0.2 %
Water	4.5 to 5.5 %	4.9 %
Heavy metals	<or= 5 ppm	Complies
Residue on ignition	<or= 0.1 %	0.1 %
Total aerobic microbial count	<or= 100/g	1 /g
Total combined molds and yeast count	<or= 50/g	1 /g
Salmonella species	Absence in 10g	Complies
Escherichia coli	Absence in 10g	Complies

CONCLUSION : Complies

**FOURNIER 1005214**

QUALITY ASSURANCE MANAGER

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12/11/96

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## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: MAGNESIUM STEARATE	
SUPPLIER . . . . .	: QUARRE MARCEL ET CIE	
SUPPLIER BATCH . . . . .	: MGS60182	
PRODUCT CODE . . . . .	: 402597	INTERNAL BATCH N° : 43605
EXPIRY DATE . . . . .	: 5/06/98	DATE OF RECEIPT : 11/06/96
SPECIFICATION . . . . .	: 01-00161/4	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristics	White ,fine ,bulky powder	Complies
Solubility	Insoluble in water, in alcohol and ether	Complies
Identification A	Responds to the test for magnesium	Complies
Identification B : GC	Retention times of stearic and palmitic acids identical to the reference solution	Complies
Acidity or alkalinity	<or= 0.05 ml 0.1N HCl or 0.1N NaOH	Complies
Chloride	<or= 0.1%	Complies
Sulfate	<or= 1.0%	Complies
Lead	<or= 0.001%	Complies
Loss on drying	<or= 6.0 %	3.5 %
Total bacterial count	<or= 1 000/g	4 /g
Yeasts and molds	<or= 500/g	4 /g
Salmonella species	Absence in 10g	Complies
Escherichia coli	Absence in 10g	Complies
Relative content of stearic acid	>or= 40 %	77 %
Relative content of stearic acid and palmitic acid	>or= 90 %	100 %
OVI : Methylene chloride	<or= 500 ppm	
OVI : Chloroform	<or= 50 ppm	
OVI : Benzene	<or= 100 ppm	
OVI : Trichloroethylene	<or= 100 ppm	
OVI : 1,4 Dioxane	<or= 100 ppm	
Assay (expressed in magnesium)	4.0 to 5.0 % (calculated on the dried basis)	4.4 %

CONCLUSION : Complies

FOURNIER 1005215

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FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

```
=====
PRODUCT . . . . . : CROSPVIDONE XL10 USP
SUPPLIER . . . . . : ISP
SUPPLIER BATCH . . . . . : N60409
PRODUCT CODE. . . . . : 402699          INTERNAL BATCH N° : 41192
EXPIRY DATE . . . . . : 12/19/96        DATE OF RECEIPT : 6/19/96
SPECIFICATION. . . . . : 01-00180/1
=====
```

TESTS	SPECIFICATIONS	RESULTS
Characters	Very fine white, tasteless powder	Complies
IR spectrum	Maxima identical to those of control spectrum	Complies
Starch coloration	No blue coloration	Complies
pH of a 1% aqueous suspension	5.0 to 8.0	6.3
Water content	<or= 5.0 %	4.1 %
Sulphated ash	<or= 0.4 %	0.0 %
Water-soluble substances	<or= 1.5 %	0.5 %
Heavy metals	<or= 10 ppm	Complies
Vinylpyrrolidone	<or= 0.1 %	0.0 %
Nitrogen (relative to dry product)	11.0 to 12.8 %	11.9 %

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

Micheline ROUSSEAU

10/04/96

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FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: COMICRONISATE FENOFIBRATE/LSNa	
SUPPLIER . . . . .	: PLASTO	
SUPPLIER BATCH . . . . .	: 97549	
PRODUCT CODE. . . . .	: 402708	INTERNAL BATCH N° : 45660
EXPIRY DATE . . . . .	: 3/12/99	DATE OF RECEIPT : 3/13/97
SPECIFICATION. . . . .	: 03-08001/9	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Caracteres	Poudre tres fine ,blanche et inodore	Complies
Identification du fénofibrate	Rf identique au temoin	Complies
Dosage du laurylsulfate de sodium	3.00 to 3.70 %	
Granulométrie laser :Médiane	5.0 to 15.0 um	
Granulométrie laser :Diamètre des particules	Au moins 90% des particules de diamètre <ou= 35um	

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

3/20/97

Micheline ROUSSEAU



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## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: PURIFIED WATER	
SUPPLIER . . . . .	: PRODUCTION FONTAINE 2	
SUPPLIER BATCH . . . . .	:	
PRODUCT CODE. . . . .	: 404900	INTERNAL BATCH N° : 1971411
EXPIRY DATE . . . . .	:	DATE OF RECEIPT : 3/10/97
SPECIFICATION. . . . .	: 01-00008/10	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characters	Clear liquid, colourless , tasteless	Complies
pH	5.0 to 7.0	6.0
Oxidisable substances	Slightly pinkish with potassium permanganate	Complies
Chloride	No precipitate for at least 15 min.	Complies
Nitrate	<or= 0.2 ppm	Complies
Sulphate	No precipitate for at least 1 hour	Complies
Ammonium	<or= 0.2 ppm	Complies
Calcium and magnesium	Pure blue colour with mordant black 11R	Complies
Heavy metals	<or= 0.1 ppm	Complies
Residue on evaporation	<or= 0.001 %	0.001 %
Bacteria	<or= 100 /ml	1 /ml

CONCLUSION : Complies

-----

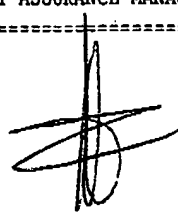
QUALITY ASSURANCE MANAGER

=====

DATE

=====  
4/11/97

Micheline ROUSSEAU



**FOURNIER 1005218**

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Protective Order**

# **Exhibit B**

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des Prés Potets  
21121 Fontaine les Dijon  
FRANCE

## ANALYSIS CERTIFICATE

=====

PRODUCT. . . . .	:	LIPANTHYL.M.200 MG.30 GEL.MV.FR
BATCH N° . . . . .	:	2177
DATE OF MANUFACTURING. . . . .	:	8/20/96
EXPIRY DATE. . . . .	:	August 1999
SPECIFICATION N° . . . . .	:	06-50002/7

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristics	Opaque, orange capsule size 1 containing a whitish powder	Complies
Desintegration	<or= 15 min	7 min
Average weight	Complies with the requirements of the European Pharmacopoeia (+/-7.5%)	Complies
Dissolution test : 20 min	>or= 75.0%	76.7 %
Dissolution test : 40 min	>or= 90.0%	91.0 %
Identification of fenofibrate	Retention time of the main peak identical to that of the reference	Complies
Assay of fenofibrate	190.0 to 210.0 mg per capsule	199.0 mg

This batch has been released by the Qualified Person in accordance with the current European GMP-requirements

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

Date

=====

9/23/96

Micheline ROUSSEAU



FOURNIER 1005202

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LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: ORANGE CAPSULES, SIZE N°1	
SUPPLIER . . . . .	: RP SCHERER	
SUPPLIER BATCH . . . . .	: 078846H010	
PRODUCT CODE . . . . .	: 406394	INTERNAL BATCH N° : 41554
EXPIRY DATE . . . . .	: 7/05/98	DATE OF RECEIPT : 7/05/96
SPECIFICATION . . . . .	: 01-00010/4	

=====

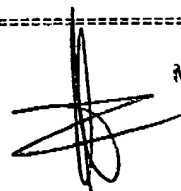
TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristics	Capsule, size n°1, orange, identical to the reference, smooth envelopé, shiny, clean, neither brittle nor fragile	Complies
Identification A	Characteristic gel	Complies
Identification B	Cloudy with picric acid	Complies
Organic colouring agent : erythrosin	Rf identical to the reference	Complies
Sulphur dioxide	<or= 0.1 %	0.0 %
Titanium dioxide	Yellow to yellow-orange colouring in the presence of hydrogen peroxyde	Complies
Ferrous oxides	Blue colouring in the presence of potassium ferrocyanide	Complies
Lubricants	<or= 0.5 %	0.3 %
Loss and drying	12.0 to 16.0 %	14.5 %
Sulphated ash	<or= 9.0 %	4.1 %
Disintegration time	<or= 15 min	11 min

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

=====  
7/19/96

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Micheline ROUSSEAU

**FOURNIER 1005203**

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Protective Order**

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: PREGELATINIZED STARCH	
SUPPLIER . . . . .	: ROQUETTE FRERES	
SUPPLIER BATCH . . . . .	: E8366	
PRODUCT CODE . . . . .	: 402100	INTERNAL BATCH N° : 40965
EXPIRY DATE . . . . .	: 12/06/97	DATE OF RECEIPT : 6/06/96
SPECIFICATION . . . . .	: 01-00162/3	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristics	A white to pale cream-coloured powder	Complies
Microscopical	Irregular, translucent, cream coloured flakes with a reticulated surface and numerous fragmented flakes	Complies
Identification	A dark blue color is produced	Complies
Acidity or alkalinity : pH of a 5% w/v dispersion in water	4.5 to 7.0	5.6
Protein	<or= 0.5 %	0.3 %
Loss on drying	<or= 15.0 %	5.7 %
Sulphated ash	<or= 0.5 %	0.2 %
Microbial contamination : Escherichia coli	1.0 g is free from Escherichia coli	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

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Micheline ROUSSEAU

=====  
7/12/96



**FOURNIER 1005204**

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LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: MAGNESIUM STEARATE	
SUPPLIER . . . . .	: FACI / QUARRECHIM	
SUPPLIER BATCH . . . . .	: MGS50623	
PRODUCT CODE . . . . .	: 402595	INTERNAL BATCH N° : 39787
EXPIRY DATE . . . . .	: 10/02/97	DATE OF RECEIPT : 4/02/96
SPECIFICATION . . . . .	: 01-00004/5	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characters	A white, very fine, light powder greasy to the touch	Complies
Solubility	Practically insoluble in water in ethanol and in ether	Complies
Identification A	Solidification point $\geq 53^{\circ}\text{C}$	54 $^{\circ}\text{C}$
Identification B	Reaction of magnesium positive	Complies
Appearance of solution S	No more intensely coloured than the control	Complies
Appearance of solution of fatty acids	Clear and not more intensely coloured than the control	Complies
Acidity or alkalinity	$\leq 0.05$ ml of 0.1N HCl or 0.1N NaOH	Complies
Acid value	195 to 210	199
Chlorides	$\leq 250$ ppm	Complies
Sulphates	$\leq 0.5$ %	Complies
Heavy metals	$\leq 20$ ppm	Complies
Loss on drying	$\leq 6.0$ %	3.0 %
Assay	3.8 to 5.0 %	4.5 %
(expressed in magnesium)		

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

5/10/96

Micheline ROUSSEAU

**FOURNIER 1005205**

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Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

```
=====
PRODUCT . . . . . : LACTOSE EFC
SUPPLIER . . . . . : SPCI
SUPPLIER BATCH . . . . . : 032/616
PRODUCT CODE . . . . . : 402552
EXPIRY DATE . . . . . : 11/14/97
SPECIFICATION . . . . . : 01-00011/4

INTERNAL BATCH N° : 40560
DATE OF RECEIPT : 5/14/96
=====
```

TESTS	SPECIFICATIONS	RESULTS
Characteristic	White to off-white crystalline powder, odourless	Complies
Solubility	Freely through slowly soluble in water, practically insoluble in alcohol	Complies
Identification A : IR spectrum	Conform to standard	Complies
Appearance of solution	Clear, odourless	Complies
Acidity or alcalinity	Colourless with phenolphthaleine ( maximum 0.4 ml NaOH 0.1N)	Complies
Specific rotatory power	54.4 to 55.9 °	55.0 °
Absorbance at 400 nm	<or= 0.04	Complies
Absorbance from 210 to 220 nm	<or= 0.25	Complies
Absorbance from 270 to 300 nm	<or= 0.07	Complies
Heavy metals	<or= to 5 ppm	Complies
Water content	4.5 to 5.5 %	5.0 %
Sulphated ash	<or= 0.1 %	0.1 %

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

6/06/96

Micheline ROUSSEAU

**FOURNIER 1005206**

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LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

```
=====
PRODUCT . . . . . : CROSPVIDONE
SUPPLIER . . . . . : ISP
SUPPLIER BATCH . . . . . : N60105
PRODUCT CODE . . . . . : 402698      INTERNAL BATCH N° : 40414
EXPIRY DATE . . . . . : 11/07/96      DATE OF RECEIPT : 5/07/96
SPECIFICATION . . . . . : 01-00006/6
=====
```

TESTS	SPECIFICATIONS	RESULTS
Characters	White or yellowish-white powder or flakes ,hygroscopic	Complies
Solubility	Practically insoluble in water, alcohol and in methylen chloride	Complies
IR spectrum	Maxima identical to those of control spectrum	Complies
Starch coloration	No blue coloration	Complies
Peroxyde	At 405 nm <or= 0.35 (400ppm)	Complies
Water-soluble substances	Résidu <ou= 75 mg (1,5%)	0.1 %
Vinylpyrrolidone	< or = 0.1%	0.0 %
Heavy metals	<or= 10 ppm	Complies
Loss on drying	<or= 5.0 %	2.7 %
Sulphated ash	<or= 0.1 %	0.1 %

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

Micheline ROUSSEAU

6/04/96

**FOURNIER 1005207**

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Protective Order**

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: PURIFIED WATER	
SUPPLIER . . . . .	: PRODUCTION FONTAINE 2	
SUPPLIER BATCH . . . . .	: 9607231	
PRODUCT CODE . . . . .	: 404900	INTERNAL BATCH N° : 9607231
EXPIRY DATE . . . . .	:	DATE OF RECEIPT : 7/23/96
SPECIFICATION . . . . .	: 01-00008/7	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characters	Clear liquid, colourless , odourless	Complies
Acidity or alkalinity	No red coloration with methyl red.No blue coloration with potassium permanganate	Complies
Oxidisable substances	Slightly pinkish with potassium permanganate	Complies
Chlorides	No precipitate for at least 15 mins.	Complies
Nitrates	<or= 0.2 ppm	Complies
Sulphates	No precipitate for at least 1 hour	Complies
Ammonium	<or= 0.2 ppm	Complies
Calcium and magnesium	Marked blue coloration with mordant black 11R	Complies
Heavy metals	<or= 0.1 ppm	Complies
Residue after evaporation	<or= 0.001 %	0.001 %

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

=====  
8/14/96

Micheline ROUSSEAU

FOURNIER 1005208

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Protective Order

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: COMICRONISAT FENO/EUROPE	
SUPPLIER . . . . .	: PLASTO	
SUPPLIER BATCH . . . . .	: 96638	
PRODUCT CODE . . . . .	: 402715	INTERNAL BATCH N° : 41740
EXPIRY DATE . . . . .	: 7/10/98	DATE OF RECEIPT : 7/11/96
SPECIFICATION . . . . .	: 03-08001/6	

=====

TESTS

-----  
Identification du fenofibrate  
Dosage du laurylsulfate de sodium  
Granulometrie laser :Mediane  
Granulometrie laser :Diametre des  
particules

SPECIFICATIONS

-----  
Rf identique au temoin  
3.00 to 3.70 %  
5.0 to 15.0 um  
Au moins 90% des particules de  
diametre <ou= 35um

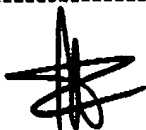
RESULTS

-----  
*{ performed by  
Synken*

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

=====



DATE

=====  
7/12/96

FOURNIER 1005209

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Protective Order

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: COMICRONISAT PENO/EUROPE	
SUPPLIER . . . . .	: PLASTO	
SUPPLIER BATCH . . . . .	: 96637	
PRODUCT CODE . . . . .	: 402715	INTERNAL BATCH N° : 41654
EXPIRY DATE . . . . .	: 7/05/98	DATE OF RECEIPT : 7/09/96
SPECIFICATION. . . . .	: 03-08001/6	

=====

TESTS

-----  
Identification du fenofibrate  
Dosage du laurylsulfate de sodium  
Granulometrie laser :Mediane  
Granulometrie laser :Diametre des  
particules

SPECIFICATIONS

-----  
Rf identique au temoin  
3.00 to 3.70 %  
5.0 to 15.0 um  
Au moins 90% des particules de  
diametre <ou= 35um

RESULTS

} performed by  
byuker

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

====  
7/11/96



FOURNIER 1005210

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# **Exhibit 13**



LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des Prés Potets  
21121 Fontaine les Dijon  
FRANCE

ANALYSIS CERTIFICATE

PRODUCT. . . . . : LIPANTIL MICRO.M.67 MG.90 GEL.MV.GB  
BATCH N° . . . . . : 77161  
DATE OF MANUFACTURING. . . . : 10/01/03  
EXPIRY DATE. . . . . : October 2006  
SPECIFICATION N° . . . . . : 06-50251/3

TESTS	SPECIFICATIONS	RESULTS
Characteristics	Yellow opaque capsule size 4, containing a whitish powder	Complies
Average mass	111.5 to 123.0 mg	114.6 mg
Mass uniformity	Complies with the European Pharmacopoeia requirements (+/- 10%)	Complies
Disintegration time	<or= 15 min	4 min
Dissolution test at 20 min	>or= 75.0 %	97.5 %
Dissolution test at 40 min	>or= 90.0%	102.9 %
Identification of fenofibrate	Retention time of the main peak corresponding to that of the reference	Complies
Assay of fenofibrate	63.6 to 70.4 mg	65.6 mg
Microbial limits (Eur.Ph.)	Periodic control	Non/No
Bacteria	<or= 1000 /g	
Yeasts and molds	<or= 100 /g	
Escherichia coli	Absence in 1g	

This batch has been released by the Qualified Person in accordance with the current European GMP-requirements

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

Date

Micheline ROUSSEAU

1/15/04

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FOURNIER 0029873

Laboratoires Fournier S.A. - Rue des Prés Potets - 21121 Fontaine-les-Dijon  
Tél. 03 80 44 78 00 - Fax 03 80 44 78 38  
Laboratoires Fournier S.A. - Siège Social - 42, rue de Longvic - 21300 Chenôve - France  
S.A. au Capital de 18 035 296 euros - RCS Dijon B 311 596 670 - TVA FR 52 311 596 670



LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

PRODUCT . . . . . : STEARATE DE MG ORIGINE VEGETALE PE  
SUPPLIER . . . . . : QUIMDIS  
SUPPLIER BATCH . . . . . : C324640  
PRODUCT CODE . . . . . : 601201 INTERNAL BATCH N° : 68084  
EXPIRY DATE . . . . . : 6/19/06 DATE OF RECEIPT : 6/19/03  
SPECIFICATION. . . . . : 01-00004/11

TESTS	SPECIFICATIONS	RESULTS
Characters	A white, very fine, light powder, greasy to the touch	Complies
Solubility	Practically insoluble in water and in ethanol	Complies
Identification C: GC	Retention times of the principal peaks in the chromatograms of the test solution and of the reference solution are approximately the same.	Complies
Identification D : magnesium	Positive reaction of magnesium	Complies
Acidity ou alkalinity	<or= 0.5ml HCl (0.01M) or NaOH (0.01M)	Complies
Chlorides	<or= 0.1 %	Complies
Sulphates	<or= 0.5 %	Complies
Cadmium	<or= 3 ppm	Complies
Lead	<or= 10 ppm	Complies
Nickel	<or= 5 ppm	Complies
Loss on drying	<or= 6.0 %	3.6 %
Assay of magnesium (calculated on the dried basis)	4.0 to 5.0 %	4.7 %
Stearic acid	>or= 40.0 %	64.5 %
Sum of stearic acid and palmitic acid	>or= 90.0 %	98.9 %
Total viable aerobic count	<or= 1 000 /g	4 /g
Escherichia coli	Absence in 1 g	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

7/15/03

Micheline ROUSSEAU

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FOURNIER 0029921

# **Exhibit 10**

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

## ANALYSIS CERTIFICATE

PRODUCT. . . . . : LIPANTHYL.M.200 MG.30 GEL.MV.TH  
BATCH No . . . . . : 73496  
DATE OF MANUFACTURING . . . . . : 10/14/02  
EXPIRY DATE. . . . . : October 2005  
SPECIFICATION No. . . . . : 06-50002/12

TESTS	SPECIFICATIONS	RESULTS
Characteristics	Opaque, orange capsule size 1, containing a whitish powder	Complies
Average mass of content	350.0 mg +/- 5 %	348.8 mg
Uniformity of mass	Complies with the requirements of the European Pharmacopoeia (+/- 7.5%)	Complies
Disintegration time	<or= 15 minutes	7 min
Dissolution test : 20 min	>or= 75.0 %	97.6 %
Dissolution test : 40 min	>or= 90.0 %	99.8 %
Identification of fenofibrate	Retention time of the main peak identical to that of the reference	Complies
Assay of fenofibrate	190.0 to 210.0 mg per capsule	200.7 mg
Microbial limits (Eur. Ph.)	Periodic control	Non/No
Bacterial count	<or= 1 000 /g	
Fungal count	<or= 100 /g	
Escherichia coli	Absence in 1g	

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

1/28/03

Micheline ROUSSEAU

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FOURNIER 0029883

Laboratoires Fournier S.A. - Rue des Prés Potets - 21121 Fontaine-les-Dijon  
Tél. 03 80 44 78 00 - Fax 03 80 44 78 38  
Laboratoires Fournier S.A. - Siège Social - 42, rue de Longvic - 21300 Chenôve - France  
S.A. au Capital de 18 035 296 euros - RCS Dijon B 311 596 670 - TVA FR 52 311 596 670

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

PRODUCT : STEARATE DE MG ORIGINE VEGETALE PE  
SUPPLIER : QUIMDIS  
SUPPLIER BATCH : 205848  
PRODUCT CODE : 601201 INTERNAL BATCH N° : 65747  
EXPIRY DATE : 6/12/05 DATE OF RECEIPT : 6/12/02  
SPECIFICATION : 01-00004/9

TESTS	SPECIFICATIONS	RESULTS
Characters	A white, very fine, light powder, greasy to the touch	Complies
Solubility	Practically insoluble in water and in ethanol	Complies
Identification C: GC	Retention times of stearic and palmitic acids identical to the reference solution	Complies
Identification D : magnesium	Positive reaction of magnesium	Complies
Acidity ou alcalinity	<or= 0.5ml HCl (0.01M) or NaOH (0.01M)	Complies
Chlorides	<or= 0.1 %	Complies
Sulphates	<or= 0.5 %	Complies
Cadmium	<or= 3 ppm	Complies
Lead	<or= 10 ppm	Complies
Nickel	<or= 5 ppm	Complies
Loss on drying	<or= 6.0 %	4.8 %
Assay of magnesium (calculated on the dried basis)	4.0 to 5.0 %	4.9 %
Stearic acid	>or= 40.0 %	66.5 %
Sum of stearic acide and palmitic acid	>or= 90.0 %	99.3 %
Total viable aerobic count	<or= 1 000 /g	4 /g
Escherichia coli	Absence in 1 g	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

6/27/02

Micheline ROUSSEAU

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FOURNIER 0029920

# **Exhibit 2**

LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des Prés Potets  
21121 Fontaine les Dijon  
FRANCE

## ANALYSIS CERTIFICATE

=====

PRODUCT. . . . .	: LIPANTHYL.M.200 MG.30 GEL.MV.FR
BATCH N° . . . . .	: 2177
DATE OF MANUFACTURING. . . . .	: 8/20/96
EXPIRY DATE. . . . .	: August 1999
SPECIFICATION N° . . . . .	: 06-50002/7

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristics	Opaque, orange capsule size 1 containing a whitish powder	Complies
Desintegration	<or= 15 min	7 min
Average weight	Complies with the requirements of the European Pharmacopoeia (+/-7.5%)	Complies
Dissolution test : 20 min	>or= 75.0%	76.7 %
Dissolution test : 40 min	>or= 90.0%	91.0 %
Identification of fenofibrate	Retention time of the main peak identical to that of the reference	Complies
Assay of fenofibrate	190.0 to 210.0 mg per capsule	199.0 mg

This batch has been released by the Qualified Person in accordance with the current European GMP-requirements

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

Date

=====  
9/23/96

Micheline ROUSSEAU

FOURNIER 1005202

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LABORATOIRES FOURNIER SA  
Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: ORANGE CAPSULES, SIZE N°1	
SUPPLIER . . . . .	: RP SCHERER	
SUPPLIER BATCH . . . . .	: 078846H010	
PRODUCT CODE . . . . .	: 406394	INTERNAL BATCH N° : 41554
EXPIRY DATE . . . . .	: 7/05/98	DATE OF RECEIPT : 7/05/96
SPECIFICATION . . . . .	: 01-00010/4	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characteristics	Capsule, size n°1, orange, identical to the reference, smooth envelopé, shiny, clean, neither brittle nor fragile	Complies
Identification A	Characteristic gel	Complies
Identification B	Cloudy with picric acid	Complies
Organic colouring agent : erythrosin	Rf identical to the reference	Complies
Sulphur dioxide	<or= 0.1 %	0.0 %
Titanium dioxide	Yellow to yellow-orange colouring in the presence of hydrogen peroxyde	Complies
Ferrous oxides	Blue colouring in the presence of potassium ferrocyanide	Complies
Lubricants	<or= 0.5 %	0.3 %
Loss and drying	12.0 to 16.0 %	14.5 %
Sulphated ash	<or= 9.0 %	4.1 %
Disintegration time	<or= 15 min	11 min

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

7/19/96

Micheline ROUSSEAU

**FOURNIER 1005203**

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Quality Assurance Department  
Rue des prés Potets  
21121 Fontaine les Dijon  
FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

```

=====
PRODUCT . . . . . : PREGELATINIZED STARCH
SUPPLIER . . . . . : ROQUETTE FRERES
SUPPLIER BATCH . . . . . : E8366
PRODUCT CODE . . . . . : 402100          INTERNAL BATCH N° : 40965
EXPIRY DATE . . . . . : 12/06/97         DATE OF RECEIPT : 6/06/96
SPECIFICATION . . . . . : 01-00162/3
=====
  
```

TESTS	SPECIFICATIONS	RESULTS
Characteristics	A white to pale cream-coloured powder	Complies
Microscopical	Irregular, translucent, cream coloured flakes with a reticulated surface and numerous fragmented flakes	Complies
Identification	A dark blue color is produced	Complies
Acidity or alkalinity : pH of a 5% w/v dispersion in water	4.5 to 7.0	5.6
Protein	<or= 0.5 %	0.3 %
Loss on drying	<or= 15.0 %	5.7 %
Sulphated ash	<or= 0.5 %	0.2 %
Microbial contamination : Escherichia coli	1.0 g is free from Escherichia coli	Complies

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

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 Micheline ROUSSEAU

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 7/12/96

**FOURNIER 1005204**

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Quality Assurance Department  
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FRANCE

RAW MATERIAL

## ANALYSIS CERTIFICATE

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PRODUCT . . . . .	: MAGNESIUM STEARATE	
SUPPLIER . . . . .	: FACI / QUARRECHIM	
SUPPLIER BATCH . . . . .	: MGS50623	
PRODUCT CODE . . . . .	: 402595	INTERNAL BATCH N° : 39787
EXPIRY DATE . . . . .	: 10/02/97	DATE OF RECEIPT : 4/02/96
SPECIFICATION . . . . .	: 01-00004/5	

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TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characters	A white, very fine, light powder greasy to the touch	Complies
Solubility	Practically insoluble in water in ethanol and in ether	Complies
Identification A	Solidification point $\geq 53^{\circ}\text{C}$	54 $^{\circ}\text{C}$
Identification B	Reaction of magnesium positive	Complies
Appearance of solution S	No more intensely coloured than the control	Complies
Appearance of solution of fatty acids	Clear and not more intensely coloured than the control	Complies
Acidity or alkalinity	$\leq 0.05$ ml of 0.1N HCl or 0.1N NaOH	Complies
Acid value	195 to 210	199
Chlorides	$\leq 250\text{ppm}$	Complies
Sulphates	$\leq 0.5 \%$	Complies
Heavy metals	$\leq 20\text{ppm}$	Complies
Loss on drying	$\leq 6.0 \%$	3.0 $\%$
Assay	3.8 to 5.0 $\%$	4.5 $\%$
(expressed in magnesium)		

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

5/10/96

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## ANALYSIS CERTIFICATE

=====  
 PRODUCT . . . . . : LACTOSE EFC  
 SUPPLIER . . . . . : SPCI  
 SUPPLIER BATCH . . . . . : 032/616  
 PRODUCT CODE . . . . . : 402552  
 EXPIRY DATE . . . . . : 11/14/97  
 SPECIFICATION . . . . . : 01-00011/4  
 =====  
 INTERNAL BATCH N° : 40560  
 DATE OF RECEIPT : 5/14/96

TESTS	SPECIFICATIONS	RESULTS
Characteristic	White to off-white crystalline powder, odourless	Complies
Solubility	Freely through slowly soluble in water, practically insoluble in alcohol	Complies
Identification A : IR spectrum	Conform to standard	Complies
Appearance of solution	Clear, odourless	Complies
Acidity or alcalinity	Colourless with phenolphthaleine ( maximum 0.4 ml NaOH 0.1N)	Complies
Specific rotatory power	54.4 to 55.9 °	55.0 °
Absorbance at 400 nm	<or= 0.04	Complies
Absorbance from 210 to 220 nm	<or= 0.25	Complies
Absorbance from 270 to 300 nm	<or= 0.07	Complies
Heavy metals	<or= to 5 ppm	Complies
Water content	4.5 to 5.5 %	5.0 %
Sulphated ash	<or= 0.1 %	0.1 %

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

6/06/96

Micheline ROUSSEAU

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RAW MATERIAL

## ANALYSIS CERTIFICATE

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=====
PRODUCT . . . . . : CROSPVIDONE
SUPPLIER . . . . . : ISP
SUPPLIER BATCH . . . . . : N60105
PRODUCT CODE . . . . . : 402698
EXPIRY DATE . . . . . : 11/07/96
SPECIFICATION . . . . . : 01-00006/6
INTERNAL BATCH N° : 40414
DATE OF RECEIPT : 5/07/96
=====
```

TESTS	SPECIFICATIONS	RESULTS
Characters	White or yellowish-white powder or flakes ,hygroscopic	Complies
Solubility	Practically insoluble in water, alcohol and in methylen chloride	Complies
IR spectrum	Maxima identical to those of control spectrum	Complies
Starch coloration	No blue coloration	Complies
Peroxyde	At 405 nm <or= 0.35 (400ppm)	Complies
Water-soluble substances	Résidu <ou= 75 mg (1,5%)	0.1 %
Vinylpyrrolidone	< or = 0.1%	0.0 %
Heavy metals	<or= 10 ppm	Complies
Loss on drying	<or= 5.0 %	2.7 %
Sulphated ash	<or= 0.1 %	0.1 %

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

Micheline ROUSSEAU

6/04/96

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## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: PURIFIED WATER	
SUPPLIER . . . . .	: PRODUCTION FONTAINE 2	
SUPPLIER BATCH . . . . .	: 9607231	
PRODUCT CODE . . . . .	: 404900	INTERNAL BATCH N° : 9607231
EXPIRY DATE . . . . .	:	DATE OF RECEIPT : 7/23/96
SPECIFICATION . . . . .	: 01-00008/7	

=====

TESTS	SPECIFICATIONS	RESULTS
-----	-----	-----
Characters	Clear liquid, colourless , odourless	Complies
Acidity or alkalinity	No red coloration whith methyl red.No blue coloration with potassium permanganate	Complies
Oxidisable substances	Slightly pinkish with potassium premanganate	Complies
Chlorides	No precipitate for at least 15 mins.	Complies
Nitrates	<or= 0.2 ppm	Complies
Sulphates	No precipitate for at least 1 hour	Complies
Ammonium	<or= 0.2 ppm	Complies
Calcium and magnesium	Marked blue coloration with mordant black 11R	Complies
Heavy metals	<or= 0.1 ppm	Complies
Residue after evaporation	<or= 0.001 %	0.001 %

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

=====  
8/14/96

Micheline ROUSSEAU

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## ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: COMICRONISAT FENO/EUROPE	
SUPPLIER . . . . .	: PLASTO	
SUPPLIER BATCH . . . . .	: 96638	
PRODUCT CODE . . . . .	: 402715	INTERNAL BATCH N° : 41740
EXPIRY DATE . . . . .	: 7/10/98	DATE OF RECEIPT : 7/11/96
SPECIFICATION . . . . .	: 03-08001/6	

=====

### TESTS

-----  
Identification du fenofibrate  
Dosage du laurylsulfate de sodium  
Granulometrie laser :Mediane  
Granulometrie laser :Diametre des  
particules

### SPECIFICATIONS

-----  
Rf identique au temoin  
3.00 to 3.70 %  
5.0 to 15.0 um  
Au moins 90% des particules de  
diametre <ou= 35um

### RESULTS

-----  
*performed by  
Synken*

CONCLUSION : Complies  
-----

QUALITY ASSURANCE MANAGER  
-----



DATE

====  
7/12/96

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FRANCE

RAW MATERIAL

ANALYSIS CERTIFICATE

=====

PRODUCT . . . . .	: COMICRONISAT FENO/EUROPE	
SUPPLIER . . . . .	: PLASTO	
SUPPLIER BATCH . . . . .	: 96637	
PRODUCT CODE . . . . .	: 402715	INTERNAL BATCH N° : 41654
EXPIRY DATE . . . . .	: 7/05/98	DATE OF RECEIPT : 7/09/96
SPECIFICATION. . . . .	: 03-08001/6	

=====

TESTS

-----  
Identification du fenofibrate  
Dosage du laurylsulfate de sodium  
Granulometrie laser :Mediane  
Granulometrie laser :Diametre des  
particules

SPECIFICATIONS

-----  
Rf identique au temoin  
3.00 to 3.70 %  
5.0 to 15.0 um  
Au moins 90% des particules de  
diametre <ou= 35um

RESULTS

} performed by  
byuker

CONCLUSION : Complies

QUALITY ASSURANCE MANAGER

DATE

====  
7/11/96

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